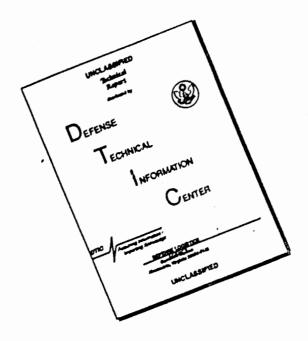
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APPENDIX A ...
LITERATURE SEARCH ABSTRACTS

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22/5/1 89203658 CA08924203658F ANALYSIS OF CARBON VERSUS RESIN AUTHOR: SZACHTA, JAMES M. LOCATION: CHEM. SYST. LAB., ARMY ARMAMENT RES. DEY. COMMAND, ABERDEEN PROVING GROUND, MD. SECTION: CA060002, CA050XXX PUBL CLASS: TECH REP JOURNAL: U. S. NTIS; AD REP. CODEN: XADRCH PUBL: 78 ISSUE: AD-A053863, PAGES: 48 PP. CITATION: 60Y. REP. ANNOUNCE. INDEX (U. S.) 1978, 78(16), 336 AVAIL: NTIS IDENTIFIERS: PINK WATER TREATMENT ADSORBENT, THT ADSORBENT PINK WATER, rdx adsorbent pink water, hmx adsorbent pink water, tetryl adsorbent pink WATER, ACTIVATED CARBON TREATMENT PINK WATER, AMBERLITE XAD4 TREATMENT PINK WATER, AMMUNITION PRODU WASTEWATER TREATMENT CR08924203658F DESCRIPTORS: ABSORBENTS; WASTEWATER TREATMENT, ADSORPTION REMOVAL PINK ACTIVATED CARBON AMBERLITE XAD4 BIOLOGICAL IDENTIFIERS: STUDIES AMMUNITION XAD 4 CAS REGISTRY NUMBERS: 118-96-7 121-82-4 479-45-8 269<u>1-41-0</u> 7440-44-0 37380-42-0 AWA 22/5/2 86184123 CA08625184123X EFFECTS OF POLLUTANTS ON EMBRYOS AND LARVAE OF FROGS: A SYSTEM FOR EVALUATING TERATOGENIC EFFECTS OF COMPOUNDS IN FRESH WATER ENVIRONMENTS AUTHOR: GREENHOUSE, GERALD A. LOCATION: UNIV. CALIFORNIA, IRVINE, CALIF. PUBL CLASS: TECH REP SECTION: CA004003 JOURNAL: AEROSP. MED. RES. LAB., (TECH. REP.) AMRL-TR (U. CODEN: 5.> AMRLD3 PUBL: 75 ISSUE: AMRL-TR-125, PAGES: 493-511 IDENTIFIERS: FROG WATER POLLUTION ANALYSIS CR08625184123X DESCRIPTORS: TERATOGENESIS: WATER POLLUTION; RANA PIPIENS: XENOPUS LAEVIS IDENTIFIERS: FROG EMBRYO ANAL BIOLOGICAL STUDIES POLLUTANTS ORG COMPDS EVALUATION POLLUTANT TERATOGENISIS 90-30-2 25619-54-9 101-67-7 302-01-2 60-34-4 CAS REGISTRY NUMBERS: 540-73-8 57-14-7

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 DETERMINATION AND MONITORING OF SOME ORGANIC EXPLOSIVES IN NATURAL AND
EFFLUENT WATER BY SINGLE-SWEEP POLAROGRAPHY
 AUTHOR: WHITNACK, GERALD C.
 LOCATION: NAV. WEAPONS CENT., CHINA LAKE, CALIF.
 SECTION: CA061002, CA050XXX, CA060XXX, CA079XXX
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  JOURNAL: IDENTIF. ANAL. ORG. POLLUT.
                                         WATER, (CHEM. CONGR. NORTH AM.
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                              ADDRESS: ANN ARBOR, MICH
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  THE EVALUATION OF THE TOXIC EFFECTS OF CHEMICALS IN FRESH WATER BY USING
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 AUTHOR: GREENHOUSE, GERALD
 LOCATION: DEP. ANAT., UNIV. CALIFORNIA, IRVINE, CALIF.
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  JOURNAL: ENVIRON. POLLUT.
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? T23/5/1-3 23/5/1 93173550 CA09318173550C CHEMICAL DETECTOR UTILIZING AN ELECTROLYTIC GEL AUTHOR: LOTZE, THOMAS H. LOCATION: USA SECTION: CA061002, CA079XXX PUBL CLASS: PAT CODEN: USXXAM JOURNAL: U.S. PUBL: 800610 PAGES: 4 PP. PATENT NO: 4207162 APPLIC NO: 18154 DATE: 790307 CLASS: 204-195R 601N27/46 ASSIGNEE: CAMBRIDGE INSTRUMENT CO., INC. IDENTIFIERS: HYDRAZINE DETH BOILER WATER APP, ELEGTROLYTIC GEL HYDRAZINE DETH APP, SILVER DXIDE GEL WATER ANALYZER CA09318173550C IDENTIFIERS: ANALYSIS DETN BOILER WATER ELECTROCHEM APP HYDRAZINE ETHERS ELECTROLYTE GEL CONTG CAS REGISTRY NUMBERS: 302-01-2 7732-18-5 9004-62-0 20667-12-3 23/5/2 93137581 CA09314137581E THE DXIDATION STATE DIAGRAM - A POTENTIAL TOOL FOR STUDYING REDOX CHEMISTRY IN SEA WATER AUTHOR: WONG, GEDRGE T. F. LOCATION: INST. OCEANOGR., OLD DOMINION UNIV., NORFOLK, VA, 23508, USA SECTION: CA061001 PUBL CLASS: JOURNAL JOURNAL: MAR. CHEM. CODEN: MRCHBD PUBL: 80 SERIES: 9 ISSUE: 1 PAGES: 1-12 IDENTIFIERS: OXIDN STATE DIAGRAM REDOX SEAWATER, NITROGEN OXIDN STATE DIAGRAM SEAWATER, MANGANESE OXIDN STATE DIAGRAM SEAWATER CA09314137581E DESCRIPTURS: REDUX REACTION; VALENCE; WATERS, OCEAN IDENTIFIERS: USES MISCELLANEOUS OXION STATE DIAGRAM DETN MITROGEN SYSTEM CHEM SEAWATER MANGANESE OCCURRENCE STUDY DIAGRAMS SYSTEMS CAS REGISTRY NUMBERS: 302-01-2 1313-13-9 1332-62-3 1336-21-6 7439-96-5 7727-37-9 7782-77-6 7803-49-8 10024-97-2 10102-43-9 10544-72-6 14333-13-2 14797-55-8 14333-14-3 14546-48-6 14797-65-0 14798-03-9

22/5/7 93116717 CR09312116717P ULTRASOUND LEVEL-METER FOR MEASURING PROPELLANT LEVELS IN THE TANKS OF ARIANE FIRST AND SECOND STAGES AUTHOR: DEMARAIS, JEAN CLAUDE; DEDM, ALAIN LOCATION: GROUPE RECH., OFF. NATL. ETUD. RECH. AEROSP., CHATILLON, FR. PUBL CLASS: JOURNAL SECTION: CA050002 JOURNAL: RECH. AEROSP. CODEN: REARAU PUBL: 80 SERIES: 194, PAGES: 9-22 LANGUAGE: FR IDENTIFIERS: PROPELLANT LEVEL ROCKET TANK CA09312116717P DESCRIPTORS: PROPELLANTS; SOUND AND ULTRASOUND, CHEMICAL AND PHYSICAL EFFECTS. IDENTIFIERS: DETH LEVEL ROCKET TANKS USES MISCELLAMEOUS VALUE CAS REGISTRY NUMBERS: 57-14-7 10102-44-0 17.71 22/5/8 93100878 CA09310100878F DETECTOR FOR FUMES OF HYDRAZINE AND ITS DERIVATIVES AUTHOR: CROOMES, EDGAR F.; MURFREE, JAMES A. LOCATION: USA SECTION: CA059003, CA079XXX PUBL CLASS: PAT JOURNAL: U.S. CODEN: USXXAM PUBL: 800429 PAGES: 4 PP. DATE: 780615 PATENT NO: 4200608 APPLIC NO: 915706 CLASS: 422-97, 601N27/02, 601N27/16, 601N31/10 ASSIGNEE: UNITED STATES DEPT. OF THE ARMY IDENTIFIERS: HYDRAZINE DETN AIR SENSOR, METHYLHYDRAZINE DETN AIR SENSOR, DIMETHYLHYDRAZINE DETN AIR SENSOR Χ CA09310100878F DESCRIPTURS: AIR AMALYSIS DETN APP USES- MISCELLANEOUS IRIDIUM CONTE DETECTORS IDENTIFIERS: HYDRAZINE DERIVS ALUMINA PELLET CAS REGISTRY NUMBERS: 57-14-7 60-34-4 302-01-2 1344-28-1 7439-88-5 parter une 22/5/9 93097901 CA09310097901A EXPLOSIVES BY NEGATIVE ION CHEMICAL ANALYSIS IDNIZATION SPECTROMETRY AUTHOR: YINGN, JEHUDA LOCATION: DEP. ISOT. RES., WEIZMANN INST. SCI., REHOVOT, 76100, ISRAEL SECTION: CA050003, CA080XXX PUBL CLASS: JOURNAL JOURNAL: J. FORENSIC SCI. CODEN: JFSCAS PUBL: 80 SERIES: PAGES: 401-7 IZZNE: S IDENTIFIERS: EXPLOSIVE ANALYSIS ANION MASS SPECTROMETRY CA09310097901A DESCRIPTORS: EXPLOSIVES; MASS SPECTROSCOPY, NEG. - ION, CHEM. - IONIZATION IDENTIFIERS: ESTERS CAS REGISTRY NUMBERS: 55-63-0 78-11-5 118-96-7 121-82-4 2691-41-0

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22/5/4 93188652 CA09320188652M TESTING PROCEDURE TO DETERMINE THE DEFLAGRATION PROPERTIES OF EXPLOSIVES AUTHOR: BIGOURD, J.; MICHOT, C. LOCATION: CENT. ETUD. RECH. CHARBONAGES; VERNEUIL-EN-HALATTE; F-60550; FR. SECTION: CA050003 PUBL CLASS: JOURNAL JOURNAL: PROPELLANTS EXPLOS. CODEN: PREXD4 PUBL: 80 SERIES: 5 PAGES: 34-6 ISSUE: 2-3 LANGUAGE: FR IDENTIFIERS: DEFLAGRATION TEST EXPLOSIVE CA09320188652M DESCRIPTORS: COMBUSTION, DEFLAGRATION; EXPLOSIVES IDENTIFIERS: ESTERS DETN USES MISCELLANEOUS COMPOUNDS CAS REGISTRY NUMBERS: 55-63-0 78-11-5 118-96-7 2691-41-0 6484-52-2 7790-98-9 22113-87-7 22/5/5 93168765 CA09318168765X HIGH SPEED GEL PERMEATION CALIFRATION PROCESSING IN AND DATA CHRUMATUGRAPHY AUTHOR: KOHN, E.; ASHCRAFT, R. W. LOCATION: DEV. DIV., MASON AND HANGER-SILAS MASON CO., INC., AMARILLO, TX , USA PUBL CLASS: JOURNAL SECTION: CA035005 JOURNAL: CHROMATOGR. SCI. CODEN: CH ISSUE: LIQ. CHROMATOGR. POLYM. RELAT. MATER. CODEN: CHESAL PUBL: 77 SERIES: 8 PAGES: 105-20 IDENTIFIERS: GEL PERMEATION CHROMATOG INTERNAL STD. MOL WT POLYSTYRENE CHROMATOS STD, DATA PROCESSING GEL CHROMATOS POLYMER, CALCH GEL PERMEATION CHROMATOS POLYMER CA09318168765X DESCRIPTORS: AIR; CHROMATOGRAPHY, GEL; DATA; MOLECULAR WEIGHT IDENTIFIERS: INTERNAL STDS HIGH SPEED PERMEATION WT DETN POLYSTYRENE POLYMERS PROCESSING RELATION CAS REGISTRY NUMBERS: 95-50-1 2691-41-0 9003-53-6 33086-17-8 22/5/6 93119554 CR09312119554U Air Deta. DETECTION OF HYDRAZINE AUTHOR: STETTER, JOSEPH R. LOCATION: USA SECTION: CA059001, CA079XXX PUBL CLASS: PAT JOURNAL: U.S. CODEN: USXXAM PUBL: 800506 PAGES: 6 PP. PATENT NO: 4201634 APPLIC NO: 916296 DATE: 780616 CLASS: 204-1T, 601N27/46 ASSIGNEE: EMERGETICS SCIENCE, INC. SECTION AIR ELECTROCHEM APP CR09312119554U DESCRIPTORS: AIR ANALYSIS IDENTIFIERS: DETECTION ELECTROCHEM APP HYDRAZINE DERIV CAS RESISTRY MUMBERS: 57-14-7 60-34-4 " William Come Hatty Hage Language

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  AUTHOR: RAULIN, F.; PRICE, P.; PONNAMPERUMA, C.
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                       PUBL CLASS: JOURNAL
  SECTION: CA080004
   JOURNAL:
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                                                   CODEN: ALBYBL
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  AUTHOR: CHEN, CHUAN-WEN; MING, CHANG-JIANG; LIU, YAC-TIAN; WANG, LIAN-JIE
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  LOCATION: CHANGCHUN INST. APPL. CHEM., ACAD. SIN., CHANGCHUN,
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CHINA
                                   PUBL CLASS: JOURNAL
  SECTION: CR080002; CA073XXX
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 DIOXIDE LASER PHOTOACOUSTIC SPECTROMETER
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   AUTHOR: FLOWERS, G. L.
   LOCATION: MASON AND HANGER-SILAS MASON CO., INC., AMARILLO, TX, USA
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                                                          ISSUE: MHSMP-80-04,
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きょりきゅうなて UAU9309094587S AMALYSIS OF INTRA- AND INTERMOLECULAR INTERACTIONS RELATING TO THE THERMOPHYSICAL BEHAVIOR OF .ALPHA.-, .BETA.-, AND .DELTA.-OCTAHYDRO-1,3,5,-7-TETRANITRO-1,3,5,7-TETRAAZOCINE AUTHOR: BRILL, T. B.; REESE, C. O. LOCATION: DEP. CHEM., UNIV. DELAWARE, NEWARK, DE, 19711, USA SECTION: CA022008, CA050XXX PUBL CLASS: JOURNAL JOURNAL: J. PHYS. CHEM. CODEN: JPCHAX PUBL: 80 SERIES: 84 ISSUE: 11 PAGES: 1376-80 IDENTIFIERS: TETRAAZOCINE TETRANITRO STABILITY POLYMORPH CR09309094587S CONFORMATION AND CONFORMERS; CRYSTAL STRUCTURE-PROPERTY DESCRIPTORS: RELATIONSHIP: POTENTIAL ENERGY AND FUNCTION; THERMAL DECOMPOSITION IDENTIFIERS: STABILITY FORMS OCTAHYDROTETRANITROTETRAAZOCINE POLYMORPHS CAS REGISTRY NUMBERS: 2691-41-0 22/5/11 93049727 CA09306049727B NUMBESTRUCTIVE CONTROL OF ELECTROEXPLOSIVE INTERFACE OF AN EXPLODED-CORD XDETUNATUR USING THE THERMAL RESPONSE METHOD AUTHOR: KASSEL, C.; CHRETIEN, N. LOCATION: CEA, SEVRAN, 93270, FR. SECTION: CA050003 PUBL CLASS: TECH REP JOURNAL: EUR. SPACE AGENCY, (SPEC. PUBL.) ESA SP CODEN: ESPUD4 ISSUE: ESA SP-144, EXPLOS. PYROTECH.-APPL. SPAT., LANGUAGE: FR 163-9 IDENTIFIERS: CORD DETONATOR EXPLOSIVE ELECTROTHERMAL ANALYSIS CA09306049727B DESCRIPTORS: DETONATORS, CORD; EXPLOSIVES IDENTIFIERS: ANAL MONDESTRUCTIVE ELECTROTHERMAL CAS REGISTRY NUMBERS: 78-11-5 PETN 22/5/12 . AIR 92202642 CA09224202642W USE OF A 6LC CONCENTRATOR TO IMPROVE ANALYSIS OF LOW LEVELS OF AIRBORNE HYDRAZINE AND UNSYMMETRICAL DIMETHYLHYDRAZINE AUTHOR: MAZUR, J. F.; PODOLAK, G. E.; HEITKE, B. T. LOCATION: U.S. ARMY ENVIRON. HYG. AGENCY, ABERDEEN PROVING GROUND, MD, 21010, USA SECTION: CA059001, CA079XXX, CA080XXX PUBL CLASS: JOURNAL CODEN: AIHAAP PUBL: 80 JOURNAL: AM. IND. HYG. ASSOC. J. SERIES: ISSUE: 1 PAGES: 66-9 IDENTIFIERS: AIRBORNE HYDRAZINE DETN GAS CHROMATOG, CONCENTRATOR LOW LEVEL DIMETHYLHYDRAZINE DETN, METHYLHYDRAZINE DETN AIR GAS CHROMATOG CR09224202642W DESCRIPTURS: AIR ANALYSIS DETH GAS LIQ CHROMATOG PRECONCENTRATOR COLUMNS HYDRAZINE IDENTIFIERS: DIMETHYLHYDRAZINE CAS REGISTRY NUMBERS: 57-14-7 302-01-2 CONTROLLER

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  DETONATION INITIATION BEHAVIOR OF SOME HMX/AP/A1 PROPELLANTS
  AUTHOR: DICK: J. J.
  LOCATION: LOS ALAMOS SCI. LAB., UNIV. CALIFORNIA, LOS ALAMOS, NM,
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  AUTHOR: PRIOR, J.
  LOCATION: DYNAMIT MOBEL A.-6., TROISDORF, FED. REP. GER.
  SECTION: CA050003
                      PUBL CLASS: JOURNAL
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  IDENTIFIERS: CONTG DETN DETONATION ENERGY
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22/5/16 92127057 CA09215127057D DETERMINATION OF DAMINOZIDE RESIDUES ON FOODS AND ITS DEGRADATION TO 1,1-DIMETHYLHYDRAZINE BY COOKING AUTHOR: NEWSOME, WILLIAM H. LOCATION: FOOD RES. DIV., DEP. NATL. HEALTH WELFARE, OTTAWA, ON, KIA OL2, CAN. SECTION: CA017002, CA005XXX PUBL CLASS: JOURNAL JOURNAL: J. AGRIC. FOOD CHEM. CODEN: JAFCAU PUBL: 80 SERIES: 28 PAGES: 319-21 ISSUE: 2 IDENTIFIERS: DAMINOZIDE GAS CHROMATOG, APPLE DAMINOZIDE DETN, COOKING APPLE DAMINOZIDE, HYDRAZINE APPLE DAMINOZIDE CA09215127057D DESCRIPTORS: APPLE; COOKING; GRAPE; PEACH; PLUM; TOMATO IDENTIFIERS: FORMATION DAMINOZIDE HYDRAZIDES DETN FOOD GAS CHROMATOG APPLES DECOMPN DIMETHYLHYDRAZINE CAS REGISTRY NUMBERS: 57-14-7 1596-84-5 L-Willitz 22/5/17 92081488 CA09210081488Q AND X ELECTROCHEMICAL DETERMINATION 0F HYDRAZINE AND METHYL-1,1-DIMETHYLHYDRAZINE IN AIR AUTHOR: STETTER, J. R.; TELLEFSEN, K. A.; SAUNDERS, R. A.; DECORPO, J. LOCATION: ENERGETICS SCI. DIV., BECTON DICKINSON AND CO., ELMSFORD, 10523, USA SECTION: CA059002, CA072XXX, CA079XXX, CA080XXX PUBL CLASS: JOURNAL CODEN: TLNTA2 JOURNAL: TALANTA PUBL: 79 SERIES: 26 :SU22I PAGES: 799-804 IDENTIFIERS: HYDRAZINE DETH AIR ELECTROCHEM CELL, METHYLHYDRAZINE DETH AIR ELECTROCHEM CELL CR09210081488Q DESCRIPTORS: AIR ANALYSIS; ELECTROLYTIC CELLS IDENTIFIERS: DETN ELECTROCHEM HYDRAZINE METHYLHYDRAZINE HYDRAZINES CAS REGISTRY NUMBERS: 57-14-7 60-34-4 302-01-2 LOWH indiane no 22/5/18 millegue 92025169 CA09204025169Z TRANSFORM INFRARED SPECTROSCOPY FOR THE HIGH-RESOLUTION FOURIER INVESTIGATION OF DECOMPOSITION GASES GENERATED BY AGING ORGANIC MATERIALS AUTHOR: HAALAND, D. M.; RÍYORD, G. E. LOCATION: SANDIA LAB., ALBUQUERQUE, NM, USA SECTION: CA050003 PUBL CLASS: TECH REP JOURNAL: REPORT CODEN: DEREPS PUBL: 79 ISSUE: SAND-79-0935C, PAGES: 6 PP. CONF-790632-6, CITATION: ENERGY RES. ABSTR. 1979; 4(17); ABSTR. NO. 45310 AYAIL: NTIS IDENTIFIERS: AGING EXPLOSIVE LONG TERM, PETN LONG TERM AGING, HAS LONG TERM AGING CA09204025169Z DESCRIPTORS: EXPLOSIVES: INFRARED SPECTRA: FOURIER-TRANSFORM IDENTIFIERS: --- AGING DECOMPN GAS PRODUCT DETN LONG TERM FOURIER SPECTROSCOPY ANALYSIS PROPELLANTS TRANSFER GASES

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TNBA Literature Sernes.

31/5/2 36026591 CA08605026591U THE EVALUATION OF THE TOXIC EFFECTS OF CHEMICALS IN FRESH WATER BY USING FROG EMBRYOS AND LARVAE AUTHOR: GREENHOUSE, GERALD LOCATION: DE ? T3/5/1-5 5/5/1 91091604 CR09111091604T 3,6-BIS-SUBSTITUTED S-TETRAZINES
AUTHOR: GUITHER, WILLIAM D.; COBURN, MICHAEL D.; CASTLE, RAYMOND N. LOCATION: UNIV. WISCONSIN, MENASHA, WI, 54952, USA SECTION: CA028022 PUBL CLASS: JOURNAL HETEROCYCLES JOURNAL: CODEN: HTCYAM PUBL: 79 SERIES: 12 122UE: 6 PAGES: 745-9 IDENTIFIERS: TETRAZINE DIPHENYL, BENZONITRILE CYCLOCONDENSATION HYDRAZINE THIOSEMICARBAZIDE PHENYL CYCLOCONDENSATION. ANILINOTETRAZINE, PHENYLFORMIMIDATE CYCLOCONDENSATION, AMINOTETRAZINE PICRYL CA09111091604T DESCRIPTORS: CYCLOCONDENSATION REACTION IDENTIFIERS: REACTIONS THIOSEMICARBAZIDE METHYLATION PHENYLFORMIMING HYDRAZIDE HYDRAZINE TETRAZINE BENZONITRILES TETRAZINES DIAMINOTETRAZINE CXIMATION DEHYDRATION REDN PREPN NITRATION CXIDATIVE DERIV ACETYLATION PICRYL CHLORIDE CAS REGISTRY NUMBERS: 74-88-4 79-19-6 88-88-0 100-47-0 302-01-2 364-44-3 619-24-9 873-62-1 874-90-8 4278-02-8P 5351-69-9 6830-78-0P SP 19617-90-4P 35600-34-1P 37841-25-1P 37932-43-7P 57508-53-9 606-34-8 14141-66-3P 19617-90-4P 35600-34-1P 37841-25-1P 37932-43-7P 57508-53-9 59995-93-6P 71123-38-1P 71123-39-2P 71123-40-5P 71123-41-6P 71123-42-7P 71123-43-8P 71123-44-9P 71123-45-0P 71123-46-1P 71123-47-2P 71123-48-3P 5/5/2 ... 91059620 - CA09108059620J THE RELATIONSHIP OF IMPACT SENSITIVITY WITH STRUCTURE OF ORGANIC HIGH EXPLOSIVES. II. POLYNITROAROMATIC EXPLOSIVES AUTHOR: KAMLET, M. J.; ADOLPH, H. G. LOCATION: WHITE DAK LAB., NAY. SURF. WEAPONS CENT., SILVER SPRING, MD, 20910, USA SECTION: CA050003 PUBL CLASS: JOURNAL MAJOURNAL: PROPELLANTS EXPLOS. CODEN: PREXD4 PUBL: 79 SERIES: 4 ISSUE: 2 PAGES: 30-4 IDENTIFIERS: NITROAROM EXPLOSIVE IMPACT SENSITIVITY STRUCTURE CR09108059620J DESCRIPTORS: EXPLOSIVES; MOLECULAR STRUCTURE-PROPERTY RELATIONSHIP IDENTIFIERS: EXPLOSION IMPACT SENSITIVITY POLYNITROAROM COMPDS CAS REGISTRY NUMBERS: 82-71-3 88-89-1 99-35-4 118-96-7P 129-66-8 131-73-7 489-98-5 519-44-8 602-99-3 6<u>06-34-9</u> 606-35-9 616-74-0 860-83-3 1150-40-9 1630-08-6 3698-54-2 4328-17-0 4433-16-3 5180-53-0 6093-29-4 6538-39-2 14185-44-5 14185-47-8 17215-44-0 21985-87-5 22167-47-1 14184-98-6 70862-23-6 70862-24-7 24577-68-2 60762-70-1 37841-25-1 -42449-44-5 70862-25-8 70862-26-9 70862-27-0 70862-29-1 70862-29-2

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5/5/3 88050419 CA08807050419F

HETEROCYCLES IN ORGANIC SYNTHESIS. VIII. SODIUM 4,6-DIPHENYL-1-OXIDO-2--PYRIDONE: REAGENT FOR THE CONVERSION OF PRIMARY HALIDES INTO ALDEHYDES AUTHOR: COOK, MICHAEL J.; KATRITZKY, ALAN R.; MILLET, GEORGE H. LOCATION: SCH. CHEM. SCI., UNIV. EAST ANGLIA, NORWICH, ENGL. SECTION: CA025015, CA027XXX, CA023XXX PUBL CLASS: JOURNAL SERIES: 7 JOURNAL: HETEROCYCLES CODEN: HTCYAM PUBL: 77 ISSUE: PAGES: 227-30

ALKOXYPYRIDONE PREPH DECOMPH, AROM ALIPH, ALDEHYDE IDENTIFIERS: HYDROXYPYRIDONE ALKYLATION, PYRIDONE ALKOXY DECOMPN

CA08807050419F

DESCRIPTORS: ALDEHYDES, PREPARATION

IDENTIFIERS: REACTION HYDROXYPYRIDONE SODIUM SALT PREPH PRIMARY HALIDES REACTIONS ALKYL THERMOLYSIS

CAS REGISTRY NUMBERS: 89-98-5P 100-11-8 100-39-0 100-52-7P 106-95-6 111-71-7P 123-72-8P 135-02-4P 591-97-9 606-34-8P 611-19-8 629-04-9 4170-30-3P 7176-28-5 7468-67-9P 22115-41-9 24964-64-5P 26478-91-1 26602-89-1 28188-41-2 52289-93-7 65218-74-8P 65218-75-9P 65218-76-0P 65218-77-1P 65218-78-2P 65218-79-3P 65218-80-6P 65218-81-7P 65218-82-8P 65218-83-9P 65218-84-0P 65218-85-1P 107-02-8P 555-16-8P

92093998 CR09211093998U

HETEROCYCLES IN ORGANIC SYNTHESIS. PART 25. REAGENTS FOR THE CONVERSION OF HALIDES INTO ALDEHYDES AND KETCHES

AUTHOR: KATRITZKY, ALAN R.; COOK, MICHAEL J.; BROWN, S. BRUCE; CRUZ, RAYMUNDO; MILLET, GEORGE H.; ANANI, ALI

LOCATION: SCH. CHEM. SCI., UNIV. EAST ANGLIA- NORWICH, NR4 ?TJ, ENGL. SECTION: CA025015, CA023XXX, CA027XXX, CA028XXX PUBL CLASS: JOURNAL JOURNAL: J. CHEM. SOC., PERKIN TRANS. 1 CODEN: JCPRB4 ISSUE: 10 PAGES: 2493-9

IDENTIFIERS: PYRIDINGNE BENZYLOXY CONVERSION BENZALDEHYDE, BENZALDEHYDE, ALKAMAL, QUIMAZOLINONE BENZYLOXY CONVERSION BENZALDEHYDE

CA09211093998U

DESCRIPTORS: ALDEHYDES, PREPARATION; ALKYL, HALIDES; ARALKYL BROMIDES; ARALKYL CHLORIDES; KETONES, PREPARATION

IDENTIFIERS: PREPH M ISOPROPOXYPYRIDONE QUINAZOLINONE INTERMEDIATES O ALKYLATION HYDROXYPYRIDONE HYDROXYQUINAZOLINONE DERIYS BENZYLDXYPYRIDONE PEACTIONS ALKENYLATION ALLYLOXYPYRIDONE BENZHYDRYLOXYPYRIDONE HYDROXYLATION REACTION SODIUM BENZYL THERMOLYSIS PHOTOLYSIS CONVERSION VIA ALKOXYPYRIDONE **ARALKOXYPYRIDONE**

67-64-1P 75-26-3 89-96-5P 98-86-2P CAS REGISTRY NUMBERS: 99-61-6P 100-11-8 100-39-0 100-52-7P 103-63-9 104-82-5 104-83-6 104-87-0P 104-88-1P 105-07-7P 106-95-6 107-02-8P 109-65-9 111-71-7P 119-61-9P 122-78-1P 123-72-8P 124-13-0P 135-02-4P 555-16-8P 585-71-7 591-97-9 606-34-8P 611-19-8 629-04-9 629-27-6 776-74-9 1212-07-3 3958-57-4 4170-30-3P 5162-44-7 5319-72-2 7176-28-5 7319-38-2P 7468-67-9P 17201-43-3 22115-41-9 5162-44-7 5319-72-2 7176-28-5 7319-38-2P 7468-67-9P 17201-43-3 22115-41-9 24964-64-5P 26478-91-1 28188-41-2 52289-93-7 65218-74-8P 65218-75-9P 65218-77-1P 65218-78-2P 65218-79-3P 65218-80-6P 65218-81-7P 65218-82-8P 65218-83-9P 65218-84-0P 65218-85-1P 67927-04-2P 67927-05-3P 67927-06-4P 67927-07-5P 67927-08-6P 67927-09-7P 67927-10-0P 72158-35-1P 72158-36-2P 72158-37-3P 72805-13-1P 72805-14-2P 72805-15-3P 72805-16-4P 72805-23-3P 72805-24-4P 72805-25-5P 72805-26-6P 72805-27-7P 72805-28-8P 72812-98-7P 72812-99-8P

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5/5/4 37161145 CA08720161145N USE OF MICELLAR SYSTEMS IN ANALYTICAL CHEMISTRY - THEIR APPLICATION TO THE SPECTROPHOTOMETRIC DETERMINATION OF SULFITE ION WITH ACTIVATED AROMATIC COMPOUNDS AUTHOR: HÍNZE, WILLIE L. LOCATION: CHEM. DEP., WAKE FOREST UNIV., WINSTON-SALEM, N. C. SECTION: CA079006 FUBL VIII. PROC. INT. JOURNAL: COLLOID INTERFACE SCI., (PROC. INT. SERIES: 5, PAGES: 425-36 CONF. > 50TH CODEN 35YRAB PUBLISHER: ACADEMIC ADDRESS: NEW YORK, N. Y AVAIL: KERKER, MILTON SULFITE DETM PHOTOMETRY, MITROAROM REAGENT SULFITE DETM; AROM NITRO REAGENT SULFITE DETN, MICELLE MEDIUM SULFITE DETN, NITROBENZENE REAGENT SULFITE DETN, NITROBENZALDEHYDE REAGENT SULFITE NITRONAPHTHALENE REAGENT SULFITE DETN CA08720161145N DESCRIPTORS: SULFITES: MICELLES: SURFACTANTS, CATIONIC IDENTIFIERS: DETN SPECTROPHOTOMETRY SPECTRUM SPECTROPHOTOMETRIC POLYNITROAROM COMPDS CAS REGISTRY NUMBERS: 99-35-4 606-34-8_606-37-1 28995-89-3 29535-21-5 64385-47-3 64426-50-2 64426-51-3 57-09-0 5/5/5 86005295 CA08601005295K REDUCTION OF 2,4,6-TRINITROBENZALDEHYDE BY SODIUM BORGHYDRIDE AUTHOR: SOKOLOVA, V. A.; BOLDYREV, M. D.; GUDASPOV, B. V. LOCATION: LENINGR. TEKHNOL. INST., LENINGRAD, USSR SECTION: CA027022, CA024XXX PUBL CLASS: JOURNAL JOURNAL: ZH. ORG. KHIM. PUBL: 76 CODEN: ZORKAE SERIES: 12 ISSUE: 7 PAGES: 1525-7 . LANGUAGE: RUSS IDENTIFIERS: REDN TRINITROBENZALDEHYDE, BENZALDEHYDE TRINITRO REDN. HEXAMEMETHANOL TRINITRO, AZAADAMANTANE METHANOL TRINITRO CR08601005295K DESCRIPTURS: REDUCTION IDENTIFIERS: REDN SODIUM BOROHYDRIDE DTO TRINITROBENZALDEHYDE REACTIONS DIFLUOROSTYRENE CHAIN LENGTHENING DIFLUOROSTRYLLITHIUMS CAS REGISTRY NUMBERS: 606-34-8 61103-61-5P 61103-62-6P 61103-63-7 61103-64-8P 16940-66-2 109-72-8 127-18-4 116-15-4 COMPOUNDS: IMPA 35DNP

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7) User 3631 2mpr81 Print 17/2/1-7 BiAt08 Files: CA Search 80-81/Vol 94(12) (See File 104) (Copr. Am. Chem. Soc.) (Item 1 of

P4014907

. phosphorus flams photometric detector response

Lazation: Kreavoy, Maurice M.; Lieng, Tai Ming
Lazation: Dep. Chem., Univ. Minneacta, Minneacolts, MM, 8648, usA
Section: CA022008 Publ Class: JOHNAL
Januaris: J. As. Chem. Soc. Coden: JACSAT Publ: 80
Section: JACSAT Brans: 10 Peges: 3118-22
Identifiers: Leatopic enchange hosoconjugate phenoxide, fractionation testepic heteroconjugate carboxylate, hydrogen host-conjugate carboxylate, potential function heteroconjugate carboxylate, jestope effect deuterium heteroconjugate carboxylate, isotope effect deuterium 014807 CA084030148070 Structures and isotopie freatienation feeters of complemes,

Location: Vys. Sh. Chemickotechnol., Pardubice, Czech. Section: CAG2GOT Publ Class: JOHNAL Journal: Chem. Prum. Coden: CPUMA Publ: 80 Series: 30 Issue: S. Pages: 236-42 Language: Czech Identifiers: LFER BIA nitrobenzene, thermal stability dinitrobenzene \$3149529 CAGGS16149529G Thermet stability of 1,3-dimitrabenzene derivatives Author: Kederabek, Vladimir: Koudelkova, Valja; Myssova,

Chamiltanioners mathed for the determination of nerogram manners of highly to the abstraction of nerogram mathers of highly to the determination of nerogram mathers of highly to the determination of presents of the continue of the feathers of the continue of the continu

92228:10 CA00226226:190
Structure-risponse relationship of gas dirematography-flame Shedmanf-fo defection to some organishmpheaphorus compounds Author: Some Samuel: Parker, George A. Leaston: Some Bee. Biv., Cham. Syst. Lab., Abardson Proving Securit, 80, 2,0010, USA. Section: CA000004 Publ Class; JOHNAL. Journal! J. Chromatogr. Coden: JCCRAN Publ: 90 Series: 168 | Seus: 3 Pages: 231-48 | Identifiers: gas chromatog organophosphorus detactor response, response flame photometric detector organophosphorus Publ Class; JOURNAL

Analysis of phosphonic solids by ion chromatography
Author: Schiff, Leon J.; Pleva, Stephen G.; Sarver, Emory W.
Location: Chem. Syst. Leb., Dep. of the Army, Aberdeen
Proving Ground, MD, USA
Section: CAOSIOC2, CAOCSXXX, CAOCSXXX Publ Class: COMP

273/46 Publ: 79 Saries; 2, Pages: 329-44
Publisher: Ann Arbor Sci. Address: Ann Arbor, Nich
Avall: Sawicki, Eugene: Mullik, Jeses D
Identifiers: phosphonic acid analysis fon chromatog,
Isopropyl methylphosphonate ion chromatog, methylphosphonate ion chromatog, ethyl
methylphosphonate ion chromatog, esthylphosphonic acid ion chromatog, urine
phosphonic acid ion chromatog

Acid-base properties of substituted phanols and carbonylic acids in mitraesthans and sate that acids in mitraesthans and sate acids in mitraesthans and sate acids in mitraesthans and sate acids. B. A.; Kashkovekaya, E. I.
Lozation: Meuchno-lasied. Inst. Org. Poluprod. Krasitelei, Mescow, USSA
Section: CA022008 Publ Class: UDUMAL
Journal: Zh. Obsich. Khis. Coden: ZGKM4 Publ: 79
Section: CA022008 Publ Class: UDUMAL
Journal: Zh. Obsich. Khis. Coden: ZGKM4 Publ: 79
Section: Appendix of Peges: 230-58 Lenguages hass
Identifiers: phanol acidity mitromethans LFER. Carboxylic

#202#124 CA0#20#03#12# Antichellomaterase properties of texic phosphero-organic compands. I. Betarainetien of kinetic persenters of antichellomaterase Author: Chome, Jerzy; Glozak, Stanislaw Location: Wojekowa Akad, Tech., Warsaw, Pol. Section: CAGOTOGO Publ Cleas: JOHNMAL, Journal: Biul: Wojek, Akad, Tech. Coden: Series: 28 Issue: 8 Pages: 143-51

29 Series: 28 leave: 8 Pages: 143-5: Language: Pol Identifiers: cholinatorae serum inhibition kinetics, organophosphete inhibition cholinasterae serum

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16) User 3631 2apr81 • • Print 16/2/1-16 Diatog Filaio4: CA Search - 77-79/Vol 91(26) (Copr. Am. Chem. Soc.) (Item

Sits2893 CADSIDEISESSAN

Gas chromstegraphic determination of phosphorus-containing
pasticide methodilites via benzylation
Author: Baughlon, Christian G.: Cook, Alasdeir M.;
Alexander, Martin
Location: Dap. Agron., Cornell Univ., Itheca. MY, 14853, USA
Soution: CADOSCOI Pub Class: JOURNAL
JOURNAL Anal. Chem. Coden: AMCHAM Publ: 79 Series:
\$1 Issue: 12 Pages: 1949-53
Identifiers: pesticide phosphorus gas chromstog

Publ: 790816 Date: 780214 Foerderung der chemilianinescence EDTA Amalysis of texto sikyl phesphates
Author: Fritsche, Dirich
Location: Fed. Rep. Ger.
Section: Cadologi Pub! Class: PAT
Journal: Ger. Offen. Coden: GMXEX fraumhofer-Gesellschaft zur Applie No: 2808046 Assignae: Fraumofer-Gesellschaft zur Angewendten Forschung e.V. Identifiers: chemiluminescence nerve acaricide, organophosphate detn <u>Chemi</u> chloride Pages: 10 pp. Language: Ger Patent No: 2806046 Applio Class: 601431/22 D1 152574

Location: Dap. Agron., Cornell thiv., Ithaca, WY, 14853, USA Saction: CAGGG13 Publ Class: JOHNAL.
Journal: Appl. Environ. Microbiol. Coden: AEMIDF Publ: 79 Saries: 37 Issue: 3 Pages: 606-9 did in tilers: phosphate sorption phosphonate 2011, bacteria phosphonate degrap phosphonate degrap phosphonate degrap phosphonate degrap phosphonate degrap phosphonate. 91001345 CA091010013459 Factors limiting bacterial Prosphate and soil binding: factors limiting bacterial degradation of fonio pheaphorus-containing pesticide metabolites Christian G.; Cook, Alasdair M.; Author: Deughton, Alexander. Mertin

Biodegradation of phesphenate toxicants yields mathems or ethems on clasvage of the carten-phespherus bond Author: Daughton, C. G.; Cock, A. H.; Alexander, N. Lecation: Dep. Agron., Cornell Univ., Ithacs, N. Y. Section: CAODOGO Publ Class: JOURNAL Journal: FERS Microbial. Latt. Coden: FMLED7 Publ: 79 ries: S issue: 2 Pages: 91-3 Identifiers: alkyl phosphonate metab Pseudomonas CA08019146658A

Modesold CADBOOBOESSAT Pesticide breakdown products: Phespherus-containing pesticide breakdown products: quantitative utilization as phespherus seuroes by becteria Author: Cook, Alacadir N.; Deughton, Christian G.; Alexander, Martin

Location: Dep. Agron., Cornell Univ., Itheca, N. V. Saction: CA010002, CA004XXX Publ Class: JUDHAL Journal: Appl. Environ. Microbiol. Coden: AEMIDF Publ: 7g Series: 36 Issue: 8 Pages: 668-72 Identifiers: bacteria organophosphate metab. phosphate organophosphate metab.

S0054301 CA09007054301A

Baselution of soid strength in tert-butyl slockel a tapercyl alockel of substituted benzeic soids, phanels, a sliphatic carbonic of soids.

Author: Chantoon! M. K., dr.; Kelthoff, I. M. Location: Dep. Chem., Univ. Minnesois, Minnesoils, Minn. Section: CA022008 Publ Class: JUMMAL JOHN CA024008 Coden: AMCHAM Publ: 79 Series of less: 1 Pages: 133-40

51 leave: 1 Pages: 133-40
Identifiers: soly product carboxylate, acidity benzoic acid
Identic acid acidity, dissocn const acid alc, acitivity
coeff acid anion. Series:

S0024517 CA05004024517H

Determination of a pelymer-sorbate interaction perameter by a sorption method
Author: Michalek, Stefan
Location: Pol.
Section: CA03012 Publ Class: JOURNAL
Journal: Zesz. Nauk. Politech. Krakov., Chem. Coden: Journal: Zesz. Nauk. Politech. Krakov., Chem. Coden: Pol. Politech.

Identifiers: solvent rubber interaction parameter

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Gas chromatographic methods for the analysis of trace quantities of all and associated compounds (demilitarization offluent brine, brine, brine, brine, brine, brine, brine, brine, taken, taken, Tisher, Tisher, L.; Stoper, Raiph J.; Parher, George A.; Sass, Samuel Location: Edgewood Araenal, Aberdean Proving Ground, Md. Section: CA004003 Publ Class: TECH RF Location: CA004003 Publ Class: TECH RF Location: Gooden Description apport Coden: Description: 28 pp. Citation: Gov. Rep. Armounce. Index (U. S.) 1978, 78(19). GB= SARIN CA08003017 H\$7N

Avail: NIIS. Identifiers: (2 detn gas chromstog

Prog.). Neet ing Monitoring the disposal of hazardous materials Author: Colburn, Educat f. Location: Colburn, Educat f. Lab. Sat. Lab. Conf. Proc. Sournal: Jt. Conf. Sers. Environ. Pollut., (Conf. Proc. durnal: Jt. Conf. Sers. Environ. Pollut., (Conf. Proc. 4th Coden: 24AVAQ Publ: 78 Pages: 489-92 Neet

Publisher: AGL Address: Washington, D. C Identifiers: chem werfare agent disposal monitoring, waste gas loxic chem disposal, incineration toxic chem flue gas

adioces: cacessicesia
A field pertable mess spectremater for monitering organia Author: Meler, Bobert W. Location: U. S. Army Environ. Hyg. Agency, Edgewood Arsenal,

Section: CADBEDC2, CAO73XXX, CAO47XXX Publ Class: JOURNAL JOAN 19. Assoc. Coden: AlMAP Publ: 78 Series: 28 Issue: 3 Pages: 23-9 Coden: AlMAP Publ: 6 Series: 28 Issue: 3 Pages: 23-9 Coden: AlMAP Publ: 6 Series: 28 Issue: 3 Pages: 23-9 Coden: AlMAP Publ: 6 Series: 29 Issue: 3 Pages: 23-9 Coden: AlMAP Publ: 78 Series: 20 Issue: 20-9 Coden: AlmaPublish: 6 Series: 6 Se

Enthalpies and entropies if ionization of 2- and 8-substituted phenois in mathemol + uster mixtures Author: Rochester, Colin H.; Wilson, David N.
Location: Chem. Dep., Univ. Nottingham, Nottingham, Engl. Section: CAG2000, CAG68XXX Publ Class: JOHGHAL, JOHGHAL, JOHGHAS, Trans. i Coden: JCFIAR, Publ: 77 Series: 73 Isaue: 4 Pages: 560-81 Identifiers: phenoi thermody as methanol, methylphenoi thermody as methanol, methylphenoi introphenoi phenoi as methanol, introphenoi as methanol, androy ionization phenoi as methanol, substituent phenoi thermody as methanol, entropy ionization phenoi as methanol, and methanol, substituent phenoi thermody as methanol, substituent phenoi thermody as methanol, transfer that phenoi as methanol, and methanol, free energy transfer nitrophenol methanol Spontaneous readitivation of acetylcholinesterase following organizations. 3. An enalysis of anomalous readitivation. 3. An enalysis of anomalous activation kinetics. 3 technology, George M.; Broomfield, Clarence A.; Steinberg, George M.; Lanks, Karl W.; Liesks, Clairs M.; Location; Bloomed Lab., Edgewood Arsensi, Aberdeen Proving Ground, Md.
Section: CACOTOCO Publ Class: UDURMAL Journal: Blochim. Bloomys Acta Coden: Backo Publ: 77 Series: 483 | Issue: 2 Pages: 312:19

Pacters governing the influence of a first hydrogen bond on the formation of a second one by the same molecule or ion Author: thysices, Pierre L. Location: Dep. Chem., Univ. Leuven, Heverlee, Belg. Section: CA022008 Publ Class: JOURNAL JOURNAL! J. Am. Chem. Soc. Cooper: JACSAT Publ: 77 Journal: J. Am. Cham. Soc. Coden: JACSAT Publ: 77 Series: 90 Issue: 8 Pages: 2578-82 Identifiers: hydrogen bond formation effect, phenol hydrogen bond equil. IR hydrogen bond phenol, mains hydrogen bond

#7:40018 CAD#7:8:403:8F Flasms dhremstegraphy of phospherus esters Author: Preston, J. M.; Karmsek, F. W.; Kim, S. H. Lodellon: Mail. Def. Headquerters, Def. Res. Establ. Ottaws. #11mms, Ont.

JOURNAL Series: deta etr 7.00 Class: Section: CAOSSOO! CAOOSXX, CAOSOXX Pub! Class: Journal: Anal. Cham. Coden: ANCHAM Pub!: 77 49 Issue: 12 Papes: 1746-50 Identifers: phosphorus ester dath air, insecticide . warfare agent dath air.

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Seio662 CAOQ415105652v

Jenization constants of phenois in mathenol + water mixtures
Author: Rochester, Colin H.: Wilson, David N.

Location: Chem. Dep., Univ. Notifighae, Autifighae, Engl.
Section: CAO2300 Publ Class: JOHNAL

JOHNAL! J. Chem. Soc., Faraday Trans, f Coden: JCFTAR
Publ: 76 Series: 72 Issue: 12 Pages: 2930-8

Identifiers: phenoi ionization substituent effect, reaction
const pheno! ionization

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of molecular formation of polarized absorption and emission apportra
of molecular forty, Gerefant Mergulico, Lent Sagiv, Jacob; Vegav,
Amanni Manur, Vehand
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Section: CAGGOOD, CAGGOXIX Publ Class: Colt. Betrout,
Section: CAGGOOD, CAGGOXIX Publ Class: Colt. Betrout
Mel. Met. Dickretian Chem. Appl. Polariz. Spectrom.
Deb. Met. P. Pages: 84-80 Newting Bate; 78
Publisher: Land thiv. Press. Address: Lund. Seed
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polyphylans cholestenone distribution, arcm hydrocarbon
hydrocarbon. We apportra polarization hydrocarbon

Grisses Castisioness Correction of instrumental time response variation with Westernia in discremental literium delorations in the mitraridist ragin. B. M.; Andieron, A. E.; Stabo, A. G. Lecetion: Div. Biol. Sci., Hall. Rec. Count. Canada, Ottowa,

Section: CA072000 Publ Glass; JOURNAL Journal Boy, Sci. Instrum. Codon: #5184K Publ: 77 Series: 40 Issue: # Puple: 1000-4 Beneiffers: Plucrescence lifeties time response variation, ratte cerrection fluorescence UV

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Print 15/2/1-15 Diatog File3: CA Search - 1972 thru 1976 (Copr. Am. Chem. Soc.) (Item 1 of 15) User 3631 2apr81

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Publ: 76 Emissis CADESISISEISE
Improved sulcasted analysis for nanogram quantities of enganquosposphoreus agains de and VX
Author: Vanishon, Dale H.; Faller, Harold L.
Location: Edgewood Areans!, Aberdeen Proving Ground, MG.
Saction: CADD-0001 Publ Class: To Journal: U. S. MITS, AD Rep. Coden: XADRCH Publ: 76Jasue: Ab-022786, Pages: 23 pp.
Citation: Gov. Rep. Announce, Index (U. S.) 1976, 76(16),

Identifiers: organophosphorus compd detn autoanalyzerd, VX detn autoanalyzer Avail: NTIS

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#8122956 CAO#\$171229665
The role of heterogenatty in the Kinetics of a surface reaction. I Infrared characterization of the adsorption structures of exprophoghenates and their decomposition Author: Kuiper, A. E. T.; Van Bokhoven, J. J. G. M.; Medema,

Location: Chem. tab., 1ND, Rijavijk, Neth.
Section: CA023003, CA004000 Publ Class: J
Journal: J. Catal. Coden: JUTLAS Publ: 76 Series:
43 Issue: 1-3 Pages: 184-87
Identifies: serim adeorption alumina IR, decompn
setty phosphonof lucridate alumina, phosphonof lucridate methyl
adeorption, toxic organizationshorus adeorption

SEOSTACH CAOSSIDORDANT
Comparison of substituent effects on dissociation and conjugation of phanols with those of carboxyllo soids in additional title, M.M-dimathylformanide, and dimathyl suifexide Author: Chan, W.M-i Kolthoff, I. M. Location: Sch. Chan, Univ. Mirrasota, Minneapolis, Minn. Section: Sch. Chan, Univ. Mirrasota, Minneapolis, Minn. Section: CAOZZOOS Publ Class: J. Miny. Chan. Coden: JPCHAX Publ: 76 Journal: J. Phys. Chan. Coden: JPCHAX Publ: 76 Jestes: BO Issue: 12 Pages: 1306-10 Jection solvent effect tonization benzoate phenol adottituent solvent effect tonization benzoate phenol enzoate phenol adottituent solvent

B4111038 CA08416111038X Beleating erganophosphorus agents using 1-phenyl-1,3,3-buts-netriene-2-cuise and oyanide Indicating Composition Author: Poziomek, Edward J.; Crabtree, Elsanor V.; Kramer,

Pel Class: P

Section: CA059002, CA080000

Location: USA

Pages: 4 Çoden: USXXAM Publ: 751007 Journel: U.S.

Patent No: 3910763 Applic No: 868,663 Date: 691003 Class: 23-2328, GOIN
Assignes: United States Dept. of the Army
Identifiers: ches warfare agent detector, phosphonofluoridate detector, phosphonofluoridate phosphonofluoridate detector, nitrobenzaldehyde phosphonofluoridate detector

Stonesory CAOS413086027F
Spontaneous reactivation of acetylcholinesterase following sorganophosphate inhibition. I. An analysis of anomalous reactivation kinetics
Author: Hovanec, Joseph W.; Broomfield, Clarence A.; Steinberg, George M.; Lanks, Karl W.; Lieske, Claire N. Location: Edgewood Arenal, Aberdean Proving Ground, Md. Section: CAOG7004
Publ Class: T
Section: CAOG7004
Section: Ab Mep. Coden: XADRCH Publ: 75
Issue: Ab -Aois562, Pages: 15 pp. State: AD-A015562, Pages: 15 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1975, 75(24), 28
Avail: NTIS deriv phosphonyl Identifiers: acetylcholinesterasa spontaneous reactivation

7507 10 .. 9 Applic No: P 23 63 652.0 Publ Class: P fen. Coden: GWXXBX Cyalia phosphinic said esters Author: Firke, Manfred; Kleiner, Hens J. Ger. Offen. CA08317147605Y Pages: 11 pp. Patent No: 2363852 731221 Class: CO7F Location: Ger. Section: CA029007 Journal: Ger.

Assignes: Hoschst A.-G. Identifiers; oxaphospholenore, cyclization phosphonete allyl

1144506 CAOB222144505J Yaxid agent leak detector Author: Fromm, Bernard W.; Silvestri, Achille; Jones, Arthur Pages: 5 700903 Patent No: 3864885 Applic No: 78,316 Date: Cleas: 23-2848, G Oin Assignes: United States Dapt. of the Army Identifiers: sefety toxic agent leak detector container Pub) Class: P Publ: 741217 Section: CAOBBOD2, CAO47000 I

Thermodynamics of hydration of 3-substituted and 3-s-disabstituted premois Author: Persons leaded H.; buchester, Colin H.; Location: Chem. Dep., Univ. Histingham, Nottingham, Engl. Location: CA022008 Publ Clers: Visitingham, Nottingham, Engl. Journal: J. Chem. Soc., Perk. of France. 2 Coden: UCPKBH Publ: 74 Issue: 13 Peppes: 1312 18 Identifiers: phenol hydration thermody substituent CAUG2 (5097483P 12097443

Asobiji CA002130003410
Resctions of tert-butyl per esters. XII. Synthesis and reactions of alkyl tert-butyl per esters. XII. Synthesis and Author: Sosnovsky, G.; Konieczyky, M.; Konieczyky, M.; Konieczyky, M.; Conieczion: Ca02007 Publ Class: J. June, Visconsin, Milwaukee, Vis. Section: CA02007 Publ Class: J. June, Pissonsin, Milwaukee, Vis. Journal: Phosphorus Coden: Phissy Publ: 74 Series: Junes: A Pegas: 286-64 Identillers: peroxy slkylphosphorate, phosphorate peroxy, phosphine trigienyl peroxyphosphorate reaction, dicyclohexylemine peroxyphosphorate reaction, pyrophosphorate

Electroreduction of nitro compaunds in disthylene glycol in a vide temperature range Author: Khitrova, L. M.; Gorbechev, S. V. Journal: Tr. Mosk. Khim, Tekhnol. Inst. Coden: Teklaf Publ: 72 Series: 79, Pages: 102-4 Language: Russ Identificat: nitro compd. electrochen platinum, redn electrochem kinetics nitro compd. benzolo acid nitro electrochem, phenol acid nitro electroredn, phenol nitro electroredn, polarization electrolytic concn nitro compd Ş Location: USSR Section: CA072007, CA087000, CA022000, CA025000 82023485 CA08204023488U

procedure for fluorated

Fluorescent defendation of some fluoring-containing phosphoroorganic substances Author: Varbastiev, M. 1.

Location: Medara Fact. Lab., Shumen, Buig.
Section: CA080006, CA004000 Publ Class: J.
Section: CA080006, CA004000 Publ Class: J.
Section: CA080006, CA004000 Publ Class: J.
Section: Section: In Pages: 1487-9
Identifiers: fluoring phosphorus org compd datn, fluoromatry fluoring phosphorus compd, samen detn, serin datn. CA08014078177J 11191000

Relation between the molecular structure of phanols and their chromatographic properties
Author: Obstandar, M. K., Gegale, V. G.
Location: Inst. Neorg. Khim. Elektrokhim., Ibilitat, USSR Section: CA02000 Publ Class; J. Coden: Sakhahi Publ: 73 Series: 71 Issue; j. Pages: 121-4 Language: Russ
Identificate: thin layer chromatog phenol, structure effect

7

PROTECTS CA077:10782785
Reactions of tert-butyl peresters. XI. Reactions of alkyl tert-butylperoxy phosphorus at all the tert-butylperoxy phosphotes. and other phosphorus esters with benzene and aluminum chievide, and reactions of dialkyl tert-butylperoxy phosphotes with phosphorus and prescions of dialkyl tert-butylperoxy phosphotes with phosphorus of dialkyl tert-butylperoxy phosphotes with phosphorus and breations of all phosphorus and aluminum chieves. When the castions Dep. Chem. Univ. Wisconsin, Milesukee, Wis. Sections: CA29007 Publ Class: J. Coden: JOCEAH Publ: 72 Journal: J. Org. Chem. Coden: JOCEAH Publ: 72 John: 37 John: 37

i.

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2) User 3631 2apr81 Print 21/2/1-2 DIALOG Filed: CA Search - 1972 thru 1976 (Copr. Am. Chem. Soc.) (Item

Location: USSR
Section: CA073003 Publ Cless; J
Journal: Opt. Spektrosk, Coden: OPSPAN Publ: 75
Series: 36 Issue: \$ Peges: 875-81 Language: Russ
Identifiers: absorption fluorescence ternary soln

Electronic states of faine radicals formed from the vacuum-ulitraviolet photolysis of ethylemisine.

Author: Kaussaki, Mashiro; Iusaki, Mashiro; Tanaka, Ikuzo Location: Dep. Ches., Tokyo Inst., Technol., Tokyo, Japan Section: CA025004 Publ Class: Jechnol., Tokyo, Japan Journal: J. Ches. Phys. Coden: JCP546 Publ: 73 Series: 99 Issue: 12 Pepss: 6238-33 Identifiers: photolysis athylenisine isino radical, energy level isino radical, electronic state imino radical.

COMPOUND: PAHs

Print 13/8/1-47 DiALBS Filed: CA Search BO-81/Vol 94(12) (See file 104) (Copr. Am. Chem. Soc.) (Item 1 of 47) User 3631 2epr81

94089369 CAO6412089358K Quantitative analysis of polyminian aromatics from diesal schemat by high performance liquid chromatography using UV photomatric detection

Author: Boussions: F.; Unger, K. K.
Location: Inst. Anorg. Chem. Anal. Chem., Johannes Butenberg-Univ., Mainz, 8500. Fed. Rep. Ger.
Section: CAGGROG2, CAGGROXX. Publ Glass: JOURNAL Journal: Anal. Chem. Symp. Ser. Coden: ACSSDR Publ: 80 Series: 3 Issue: Recent Bev. Chromatogr. Electrophor. Pages: 238-46
Identifiers: diesel exhaust gas sepn, polynuclear arom detn

disset exhaust

CA09412089359K

Descriptors: Exhaust gases, diesel; Sas analysis Identifiers: dein 114 chroastog UV spectrosetry polynuclear

CAS Registry Numbers: '80-32-8 85-01-8 128-00-0 191-24-2 182-97-2 183-38-5 188-85-0 205-82-2 205-99-2 205-44-0 207-08-9 217-59-4 218-01-9

94068207 C408410088207U Microenalysis of polymolear aromatic hydrocarbons in

1

Author: Matawahita, Hidatawu Location: Dep. Community Environ. Sci., Mati. Inst. Public Health, Tokyo, 108, Japan Section: Codoli, CAOSSXX, CAOSOXX Publ Class: JOURNAL Journal: Prepr. Pap. - As. Ches. Soc., Div. Fuel Ches. Codon: AGFPAI Publ: 78 Series: 24 Issue: 1 Pegas: Identifiers: area hydrocarbon chromatog petrolaum, polycyclic area hydrocarbon chromatog, dual band thin layer chromatog

CA084 10068207U

Descriptors: Aromatic hydrocarbons,polycyclic,analysis; Cromatography, thin-layer;Gasoline;Patroleum products Identifiers: detn dash bands CAS Begistry Mambers: 80-32-8 88-85-3 128-00-0 191-24-2 191-26-4 198-86-0 208-44-0 207-08-9 218-01-9

Author: Hogsmenn, R.
Locaton: Lab. Spectrom. Massa, CEA-Sacisy, Sacisy, Fr.
Section: Cabbooo, Cadelxix, Cadelxix Publ Cless: JOURIAL
Journal: Pollut. Assos. Coden: Pollut. Assos.
ries: 85, Pages: 21-6 Language: Fr 94035159 CAO94080384592 Trace analysis of polynuolear arematic hydrocarbons automobile emissions

Identifiers: review polycyclic area hydrocarbon detn, exhaust polycyclic area hydrocarbon review

7- 24-4

hydrocarbons, polycyclic, analysis; Aromatic

CA0840E0354592 R
Descriptors: Aro
Exhaust gases
Identifiers: detn

B4020132 CA09404020132P
Determination of polymediaer arountic hydrocarbons in vater Author: Groeby, M. I.; Hunt, D. C.
Location: Lab. Gov. Chem., Dep. Ind., London, SE 1 840, Engl. Section: CA081002, CA0793XX, Publ. Cless: JOURNAL JOURNAL JOURNAL AND Froc. (London) Coden: ANROI Publ: B0 Series: 17 Issue: 8 Pages: 381-4 Identifiers: polymuclear arom hydrocarbon data water

Descriptors: Aromatic hydrocarbona,polycyclic,analysis identifiers: dein river water extn high performance liq chromatog CAS Registry Numbers: 50-32-8 191-24-2 103-39-5 205-99-2 206-44-0 207-08-8 7732-18-5 CA09404020132P

94018017 CAOS404018017M Quantitative analysis of polymusiesr aromatic hydrocarbons in liquid fusis

Author: Parr, Jarry L.
Location: Radian Corp., Austin, TX, USA
Section: CA081009, CA088XXX Publ Class: TECH REP
JOURNAL: Report Coden: DBREP4 Publ: 80
EPA-600/2-80-068: Grder No. PB80-187388, Pages: 44 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1980, 80(18).

hydrocarbon scentifiers: polycyclic arcm detn fuel, polycyclic arcm fuel AVAIL: NTIS

CA09404018017M

Descriptors: Fuels, dieselifuels, jet aircraft; Gasoline Identifiers: occurrence liq hydrocarbon polycyclic arom hyrocarbons CAS Registry Numbers: 80-32-8 56-55-3 85-01-8 120-12-7 128-00-0 191-24-2 192-97-2 205-44-0 217-59-4 218-01-9

47) User 3631 2apr81 • DIALOG File4: CA Search 80-81/vol 94(12) (See file 104) (Copr. Am. Chem. Soc.) (Item

CA0832220724 IV

Identification and quantification of polynuclear aroustic compounds in Synthell by rece-temperature phosphorimatry Author: Vo-Dinh: Tuan; Gammage, R. B.; Martinaz, P. R. Location: Health Sef. Res. Div., Dak Ridge Nett. Lab., Dak Ridge, IN. 37830, USA
Section: CAOSIO29, CAOROXIX Publiciass; JOUGHAL
JOURNAL Anal. Chim. Acta Coden: Acta Roder, Publ: 80
Section: 18 Saue: 2 Pages: 313-23
Identifiers: cost itq aros phosphorimatry

CA08322207241V

Descriptors: Arcastlo cospounds:Arcastlo hydrocarbons,analy-sta;Cosi, liquefled

Identifiers detn Syntholia phosphoriaatry coapds liqs polymoleaer Syntholia process CAS Registry Mambers: 90-22-8 85-01-8 86-73-7 128-00-0 206-44-0 218-01-04-2 7861-83-5 7786-17-5

Journal: Anst. Chee. Coden: ANCHAM Publ: 60 Series: 82 Issue: 13 Pages: 2083-8
Identifiers: Spoinwales area hydrocanco nausysis fluorescence, bolymolesar area hydrocarbon, selectivity fluorescence polymolesar area hydrocarbon, chromatog polymolesar area hydrocarbon; ploymolesar area hydrocarbon; ploymoles

CAO83 18 179 100C

hydrocarbon

Descriptors: Arcestic Pydrocarbona.polycyclic.analysis: Chromatography, column and liquid, high-performance; Chromatography, gas; fuorescence; Petrolaus; Specificalsis analysis; fluorescence; Cartellaus; Specificalsis analysis; fluorescence; Cartellaus; Specificalsis analysis; fluorescence; Cartellaus; Specificalsis analysis; florescence; Cartellaus; Specificalsis; Specificalsis

92159566 CAO92161596690 Studies or the polymenter areastic hydrocarbons in cruda Studies on the polymenter programmed for the selection of the

Publ Class: JOURNAL Delgakko, Del-2-bu Pages: pollutant water Section: CAO61001, CAO61XXX, CAO60XXX Publ Class:
Journal: Kenkyu Hokoku - Kaijo Hoan Dalgakko,
Coden: KHDNAR Publ: 80 Series: 28 Issue: 2
1-9 Lenguage: Jepan Meeting Date: 79
Identifiers: fingerprinting petroleum pollutant chrometog

CA09316 155566H

Descriptors: Aromatic hydrocarbons.polycyclic,biological studies;Chromatography,gas;Fuel oll;Petroleum products; effectious;Mater pollution affections olls contg (ingerprinting polynuclear crude heavy pollutant apills

Mathods of analysis for pelymiclear aromatic hydrocarbons in environmental samples are pelymiclear aromatic hydrocarbons in Author: Bancirov, R. J.; Searl, T. D.; Brown, R. A. Location: Exxon Res. and Eng. Co., Linden, NJ, 07036, USA Section: CAOO4000 Publ Class: JOURNAL OURSALS CHAN. Soc. Coden: Account Publ: Pespr., Div. Pet. Chan. Am. Chan. Soc. Coden: ACPCAI Publ: 78 Series: 23 Issue: 3 Pages: BSS-69 Identifiers: review environment arom hydrocarbon analysis

CA08315143826N . R Descriptors: Aromatic hydrocarbons.polynuclear.analysts; Environment Identifiers: detn environmental samples methods

93134744 CA093141347442
Analysis of polynuclear arematic hydrocarbons at trace lavel
in white ells by liquid chromategraphy and UV
spectral lucrimity.
Author: Colin, J. M.; Vion, G.
Location: Cent. Rech., Co. Fr. Reffinege, Harfleur, 76700,
fed. Rep. der.
Section: CA051011, CA060XXX Pub Class: JULHALL
JULINIA: Analusis Coden: AAKSCY Pub): 80 Series: B
faune: G. Peges: 24-9 Language: Fr
Identifiers: white oil polynuclear arce, chromatog
spectrofluorimetry white oil

CA083141347442

Descriptors: Arcmatic hydrocarbone,polycyclic,analysis; Hydrocarbon oils_white oils Identifiers: dath polymelear CAS Registry Numbers: 80-32-8 \$6-88-3 85-01-8 129-00-0 191-26-4 198-85-0 206-44-0 218-01-9

I

#3160760 CA0#3101607605 in high performance liquid ebrematography analysis of polymelear arematic hydrocarbons weing terrary analysis of polymelear arematic hydrocarbons weing terrary analysis of polymelear arematic hydrocarbons weing terrary abusing Sarties and they Barry B.

**Lecation: Ballinky, Barry B.

**Lecation: Ballinky, Barry B.

**Lecation: Ballinky, Barry B.

**Lecation: Candidate Comp. Saf. Health, Cincinnati, OH, 4828.

2 MXX Publ Class: JOHNAL.
-. Coden: ACSMCB Publ: 80
Anel. Tech. Occup. Health Chem. Series: 120 Issue: Anal. Tech. Occup. Health Cr Pages: 140-68 [dentifiers: pelynuclesr area hydrocarbon detn chromatog į ACS Symp. Sal

CAGE 3 10 100 700 5

Descriptors: Aromatic hydrocarbona,polycolic,analysis; Chromatography,column and liquid, high-performance;Exhaust genes;Edesco meda and seeking lidentifiers: data engine arhausts occupations ternary selvent system polymicien [19]

EJ100685 CA08310100855F Cerrelation between the concentrations of polynuclear greatic hydrocarbons and those of particulates in an urban

Ather: Harde, Takashi: Kato, Yoshihiro; Yasamura, Takaki;
Lacation: Fac. Sule. Kyo.
Location: Fac. Sci. Sci. Mniv. Tokyo, 162, Japan
Location: CAGBEOG. CAORXIX. Pub. Cleas: JAURAAL
JOURNAL: Explor. Sci. Technol. Coden: ESTHAG Publ: 80
Series: 14 Issue: 4 Pages: 416-22
Identifiers: polymucleer area hydrocarbon sir perticulate

3

CA083 10 100655F

Identifiers: data air correlation conca particulates urban ana polyaros particulate
CAS Segistry Aumbers: 80-32-8 86-88-3 129-00-0 191-24-2
196-86-0 218-01-0 Descriptors: Analysis: Aromatic hydrocarbons, polynuclear, analysis:Particles

92088175 CA08208081780

When of an equate alcellar mabile phase for separation of phases and polymostar arematic bydroathous via IPLC
Author: Arestron; Daniel W.; Henry, Susan J.
Location: Dep. Chem., Georgatoun Univ., Washington, DC, 20087, USA
Section: CAOROGOO Publ Class: JOHENAL
JOURNAL J. Liq. Chromatogr. Coden: JC:08 Publ: 80
Section: CAOROGOO Publ Class: AGGOO Publ: Banal Section: Access and Chromatogr. Banal Section: Coden: JC:08 Publ: 80
Section: Canalis mobile phase in chromatogr. Bodium dodecy: Sulfate mobile phase, phase; if chromatogracial dodecy: Wydrocarbon liq chromatogracial eluent, Pydrocarbon liq chromatogracial

arom hydrocarbon liq chromatog

Chromatography, column and liquid, high-performance; Ricelles; Phenola, analysis | Phenola, analysis | Identifiers | sodium | Anderson | Identifiers | Identif

uses miscellaneous CLS Registry Numbers: 88-68-7 88-75-5 88-89-1 91-20-3 95-48-7 100-02-7 108-46-3 108-88-2 120-12-7 120-80-9 123-31-9 181-21-3

Column-Induced selectivity in separation of polynuclear amounted by drocerbons by reversed-phase, high-performence liquid chromatography (a transpraphy Author: Colsside, A. t.; MacDonald, J. C. Location: Dep. Anal. Chem., Univ. Stockholm, Stockholm, S-tof et, Sued.
Section: CAGGOOD Publ Class: JOURNAL JOURNAL JOURNAL Chromatographia Coden: CHROST Publ: 80

Series: 13 Issue: 6 Pages: 350-2
Identifiers: 14 Chromatog polymuclear arcm hydrocarbon, reversed phase 14 chromatog column, high performance 14 chromatog column, column induced selectivity 14 chromatog manufacturer variation column selectivity chromatog

CA09308088 174P

hydrocarbons, polynuclear, analysis; and liquid, high-performance, Descriptors: Aromatic Chromatography, column reversed-phase

identifiers: maps variations columns different manufacturers relation selectivity CAS Registry Nambers: 80-32-8 83-70-3 56-56-3 191-07-1

CAOSOOTOS2649X Descriptors:

hydrocarbone, polycyclic, analysis; Aromet to

Identifiers: detn environmental samples polynuclear

01ALDS File4: CA Search 80-81/Vol 84(12) (See file 104) (Copt. Am. Chem. Soc.) (Item is of 47) User 3631 2ept81

Set to be directed to be desired with the first of the part of the property of the part of

\$2082754 CA083060827548
The vorking environment in the aluminum industry. Sampling for palynumines environments by an alumine fluidized-bad sampler Author: Gloersein, Diav. Krohn, Conrad; Toogersen, Svain; Fjeldested, Per Einer Location: Appl. Ches. Div., SIMTE, Trondreis, Horway Section: CA085002, CA0864XX, CA078XXX Publ Cless; Journal Location: CA085002, Pages: 117-24
Journal: Electrochie, Acts Caden: ELCAAV Publ: BO Series: 25 lasus: 2 Pages: 117-24
Johntflers: aluminum industry working stm. sampler air pollution aluminum industry, polynuclear arom sampler aluminum industry.

CA093080627946

Descriptors: Air analysis; Aromatic hydrocarbona, polynuclear-, analysis; Fluidized beds and systems; Sampling apparatus , identifiers: data working at a blushum industry filter alumina sampler menule aroms fluidizer contig CAS Registry Numbers: 60-32-8 56-58-3 83-32-9 85-01-8 86-73-7 80-12-0 81-20-3 91-87-6 22-85-4 120-12-7 126-00-0 132-84-9 132-56-0 191-24-2 182-92-183-34-6 185-18-01-9 206-98-2 206-44-0 207-06-9 200-96-6 217-59-4 218-01-9

93092679 CAOR306082578K Amilyas of coke even effluents for polymolear aromatic compounds

4

Safety 40VXAQ Author: Tucker, Sessel P.
Location: Robert A. Taft Lab., Netl. Inst. Occup. 1
Health, Cincinnati, Mr. 48226, USA
Section: CADSHOOD, CADSHAX Pub. Class: CDNF PROC.
Journal: Anel. Nethods Cost Cost Prod. Coden:
Publ: 79 Series: 3 Pages: 183-87
Avell: Ner: Academic Address: New York, N. V
Avell: Ner: Clarence, Jr
Identifiars: review pelymuclear area compd analysis.

offluent analysis review

COke

CA08306052579K

CAGGOGGESTEK R POSTORIA PROCESSORIA PROCESSORIA POSTORIA. - PROCESSORIA PROSTORIA PROSTORIA PROCESSORIA PROCESSORI

Scorect chosposodestan Birect determination of polynucian prometic hydronarbone in coel liquide and shale oil by laser encited Expol'miti Author: Vang. Van; B'Silva, Arthur P.; Fassel, Velmar A.; Eles, Malvarn Lecation: Dep. Cham., Joue State Univ., Ames. IA, 80011, USA Section: CAGEGOI, CAGEGUX Publ Class: JOURNAL

Series: COS 119 62 Issue: 8 Pages: 1360-1 Identifiers: arcs hydrocarbon detn lusinescence, arcs hydrocarbon, shale oil arcs hydrocarbon - Topa Journal: Anal. Ches. Coden: ANCHAM Issue: 8 Pages: 1360-1

CA08306049844N

Descriptors: Aromatic hydrocarbone,polynuclear,analysis; Coal,11q.;Hydrocarbon oils;Shale oils Identifiers: datn liqs luminascence laser radiation induced

9009468 CA0800408458W Application of rank annihilation to fluorescent willicomponent mixtures of polymolesr arematic hydrocerbons

20.28 Author: No. C. N.; Christian, G. D.; Davidson, E. R. Location: Dep. Ches., Univ. Washington, Seattle, MA,

Series: identifiers: rank annihilation fluorescence dat quantitation, polynuclear arom hydrocarbond datn fluoremetry Section: CA080006 Publ Class: JOURNAL JOURNAL Chem. Coden: AMCHAM 62 Issue: 7 Pages: 1071-9 Cdent Amchilation (1907)

CA09304036455M

Descriptors: Archatic hydrocarbons.polynuclear.analysis; Spectrochemical analysis, fluorometric; Statistics and Statistical analysis; rank annihilation in the construction and statistic identifiers: detn fluorometry quentation multicomponent pactral data quantitation apactra sixts

CAS Segistry Munders: 82-24-0 120-12-7 198-85-0 206-44-0 218-01-8 2063-00-1

93030996 CA093040309988 A comparison of the collection and A comparison of two techniques for the collection and yste of polymentear aromatic compounds in smblent air Author: Lindgren, James L.: Krauss, Henry J.: Fox, Marys

JPCAAC Location: Texas Air Control Board, Austin, TX, USA Section: CA059002 Publ Cleas: UDURAL Control Assoc. Coden: UPC July: 80 Series: 30 Issue: 2 Pages: 166-8 Identifiers: polynuclear area air absorbent polyurathana

CA0830403088R

Descriptors: Absorbents; Afr analysis; Arometic hydrocarbons, - polycyclic, occurrence; Uruthane polymers, uses and miscallaneous Identifies: polymolear compde relation collection absorbance.
CAS Registry Mambers: \$9800-31-6

DIALOG Filet: CA Search BG-B1/Vol 94(12) (See file 104) (Copr. Am. Chem. Soc.) (Item. 21 of 47) User 3631 2mp#1

Portable fluoremetric monitor to detect polynolesr arounted bydrocarbon contamination of work area surfaces duthor: Schureako, D. D. Localion: Oak Ridge Net, D. Saction: CA066001, CA062XXX, CA078XXX Publ Class: TECH

Journal: Report Coden: DOMEP3 Publ: 79 Issue Citalion: Energy Res. Abstr. 1979, 4(23), Abstr. No. 54101 Avstl: NTS Journal: Report CONF-790858-2, P

Identifiers: polynuclear area hydrocarbon dain fluoremeter. cost conversion surface contacination fluoremeter

CA08302012472P

Descriptors: Aromatic hydrocarbons,polynuclear,analysis;Coal Identifiers: dein-surface clean areas conversion portable fluorometer surfaces

#220#57% CAO#22#520##55K Mathedology for the testation of polynusian aromatic hydrocarbons for qualitative, quantitative, and blosssay shudion Chortyk, D. T.
Location: Tob. Lab., Sci. Educ. Adm., Athena, GA, 30604, USA
Section: CA004000 Publ. Class. UDIRNA.
Johnnal: Environ. Pages. Sci. Res. Coden: EVSRBT Publ: 80
Series: 16 Issue: Hydrocarbona Halogenated Hydrocarbona
Aquat. Environ. Pages: 91-108 Meeting Date: 78
Identifiers: review area hydrocarbon isolation data Author: Severson, B. F.; Snook, M. E.; Arrendale, R. F.;

Ca00228300878K R Bescriptors: Aromatic hydrocarbons, polynucless, analysis Identifiers: isolation relation

#220#50 CA0#22420#50# Ushantien of sensitized fluorescence for polynuclear artenatic Widenarten delection Author: Saith, T. R. Location: Def. Space Syst. Group, TRV, Redondo Beach, CA,

Journal: Repart Coden: D08EP4 Publ: 79 Issue: EPA/600/7-79/207; Order No. P880-108478, Pages: 47 pp. Citation: Gov. Rep. Armounce. Index (U. S.) 1980, 80(5), 770 Avail: WIIS Section: CAGGLOGG, CAGGIXXX, CAGGEXXX Publ Class: TECH MEP

Identifiers: fluorescence detection polymolear arom hydrocarbon, and test polymolear arom hydrocarbon, combustion of fluoris screening arom hydrocarbon, ges chrosetog polymucieer arom hydrocarbon, mass spectrometry polymucies

area hydrocarbon

CA09224208550M

Descriptors: Aromatic hydrocarbons.polynuclear.analysis; Chomatography.gas;Combuston gases;Sass spectroscopy Identifers: detn effluents fluorescent spot test screening samples combined fluorescence sample prior

9230262 CA092242026234
A Comparison of two techniques for the collection and analysis of polymenter arecastis compounds in ambient air Author: Lindgren, Jeses L.; Krauss, Henry J.; Fox, Marye

Location: Texas Air Control Board, Austin, TX, USA Section: CA059002, CA079XXX Publ Class: UDWHMAL Journal: Proc., Annu. Mest. - Air Pollut. Control Assoc. Coden: PRAPAR Publ: 78 Series: 71st. Vol. 2, Pages: 78-25.4, 14 pp. Arse

Identifiers: Bondapak polynuclear arom hydrocarbon sampling, polynuclear arom hydrocarbon sampling, atmospyruclear arom hydrocarbon sampling

Peges

CA08224202682J

Descriptors: Air analysis; Arosatic hydrocarbons, polycyclic, analysis; Urethanh polymers, uses and miscallaneous in Contificars: sample app collection dain sampling absorbants an Bondspak Cis polyurethans
CAS Registry Numbers: 89800-31-6

#2168936 CA09220188836H Amblysis of polymolear aromatic hydrocarbons in amiliarmental waters by high-pressure liquid chromatography Author: Sorrell, R. K.; Reding, R. Location: Off. Drinking Water, EPA, Cincinnati, OH, 45286, 25

Section: CAD61002, CAD78XXX Publ Class: JOURNAL Journal: J. Chrostogo. Goden: JOCKAN P. Series: 188 Issue: 1 Page: 656.70
Identifiers: polynuclear area hydrocarbon dein water

CA09220168936H

chromateg UV 6.5 Registry Numbers: \$6-32-8 \$3-70-3 \$6-56-3 85-01-8 120-12-7 120-00-0 181-24-2 182-87-2 183-39-8 188-56-0 205-99-2 206-44-0 207-08-9 218-01-9 2381-21-7 7732-18-5 Descriptors: Arosatic hydrocarbons,polymolear, analysis Identifiers: detn drinking natural vater high pressure liq

47) User 3631 2apr81 26 0/ DIALDG Filed: CA Search 80-81/Vol 94(12) (See file 104) (Copr. Am. Chem. Soc.) (Item

Material CAOSISISTEM of polymerless are the hydrocarbons in water and vestimation of polymerless and vestimation by a gas chromategraphic-ultraviolet appearing by a gas chromategraphic-ultraviolet appearing by a gas chromategraphic-ultraviolet appearing by K.; Brown, B. A. Author: Searl, T. D.; Bobbins, W. K.; Brown, R. A. Author: Caosino Searlion: CAOSIGO2, CAOSONIX, CAOSONIX Publ Class: TECH

Journal: ASTM Spec. Tech. Publ. Coden: ASTTAB Publ: 79
Issue: SIP 686, Meas. Org. Pollut. Vajor Mastewater,
Pages: 164-80 Measting Date: 78
Identifers: area polycyclic hydrocarbon dein wastewater,
gas chromatog UV apectrometry

Descriptors: Arometic hydrocarbone,polynuclear,analysis Identifiers: datn.gas chrometog UV spectrophotometric water CAS Registry Aumbers: 7722-18-5

92:16:133 CA082:14:16:1332
Determination of polymenteer aromatic hydrocarbons in refinery water streams
Author: Preston, H. G.; Macaluso, A.
Location: Preston of Port Aribur, TX, 77840, USA
Location: CAOS:002, CAOS:XXX, CAOSOXXX Publ Class: TECH

Journal: ASIM Spec. Tech. Publ. Coden: ASITAB Publ: 79 Issue: STP 646, Mass. Grg. Pollut. Water Mastewater, Pages: 162-63 Mesting Date: 78 Identifiers: polymoiser area hydrocarbon wastewater refinery, 114 chromatog polymoiser area hydrocarbon, reverse concn polymoiser area hydrocarbon.

CA082141161332

Descriptors: Aromatic hydrocarbons,analysts Identifiers: polynuclear dath petroleum refining wastewater CAS Registry Numbers: 7722-18-5

Selisize coosistisizes

Sevelopment of an aqueous polynuclear aromatic hydrocarbon standard reference makerial
Author: May, W. E. Brown, J. M.; Chesler, S. N.; Quenther, F.; Hilpert, L. R.; Hertz, H. S.; Wise, S. A.
Location: Cadeloo: Stand., Washington, DC, 20234, USA
Section: Cadeloo: Aromat. Hydrocarbone, Int. Syap. Chem. Unr. no. Carcinog. Mulagen., 3rd Coden: 41WSAL Publ: 79
Pages: 41:-18 Healing Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich Avall: Jones, Peter W.; Laber, Philip
Identifiers: arom hydrocarbon std ref material

CA09214116127A

Descriptors: Aromatic hydrocerbone,polynucleer.preparation identifiers: analysis atd ref material environmental assessment preprint Centification CAS Registry Numbers: 7722-16-8

92115928 CA09214115928A
Quentitative analysis of selected PAH (polymolear aromatic
dentitative analysis of selected PAH (polymolear aromatic
hydrocarbone) in aqueue effluent by high-performance liquid
chromatography
Author: Wilkinson, Johan E.; Strup, Paul E.; Jones, Peter W.
Location: Battelle-Columbus Lab., Columbus, OH, 43201, USA
Section: CA060003, CA078XXX Publ Class: COMF PROC
JOURNAL! POLYMOL! Aromat. Hydrocarbons. Int. Symp. Chem.
Biol. - Carcinog. Mattagen., 3rd Coden: 4195A. Publ: 79
Pages: 217-29 Meeting Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich
Avall: Jones, Peter W.; Laber, Philip
Identifiers: polymolear arom hydrocarbon detn westewater

CA09214115928A
Descriptors: Arcmetic hydrocarbons.polynucleer, analysis
Identifiers: dein wastewater extn 11q chromatog fluorescence

CAS Registry Wambers: \$3-70-3 91-20-3 120-12-7 206-44-0 216-01-9 7732-18-8

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47) User 3631 2apr81 8 DIALDS Filed: CA Search 80-81/Vol 84(12) (See file 104) (Copr. As. Chem. Soc.) (Item

PAN (polymenter presents bydrocarbons) emissions from a practition-charge valuation without exidation catalysts sampling and analysis valuation.
Author: Loo, Frank S. C.; Frater, Y. J.; Forris, F. Lecation: Sci. Res. Lab., Ford Not. Co., Dearborn, NI, 48121

Section: CACGEGO2, CACGECIAN Publ Class: CDNF PROC Journal I Polymel. Acoust. Pydrocarbone, 1nt. Symp. Chem. Giol. - Carding, Mategon., 3rd Coden: 4195AL Publ: 78 Pages: 83-140 Meeting Bets: 78 Coden: 4195AL Publ: 79 Publisher: Ann Arbor Sel. Address: Ann Arbor. Mich Avell: Jones, Peter W. I Labor. Philip Identifiers; polymuclear area hydrocarbon exhaust gas

CA08214118549C

CA08214115604J

7

des chramatographic seperation of high-molecular polynumiaar aromatic hydrocarbons in ampies from different sources, using temperature-stable glass capillary columns. Author: Stemberg, Ulf; Aleberg, Tomas; Blomberg, Lars; Westran, Thomas. Location: Dep. Anal. Chem., Univ. Stockholm, Stockholm, Publiciass: COM Section: CAGGGOD: CAGGGXXX, CAGGGXXX

Journal: Polymuci. Aromat. Mydrocarbona, Int. Symp. Chem. Haloi. - Carcinog, Mutagen., Ird. Coden: 41954. Publ: 79 Pape: 313-36. Meating Date: 78 Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich Address: Ann Arbor, Bich

Avail: Jones, Peter V.; Leber, Philip Identifiers: polynuclear area hydrocarbon gas chromatog, enhaust gas polynuclear area hydrocarbon, combustion gas polynuclear area hydrocarbon

Descriptors: Air analysis; Arometic hydrocarbons, polynuclear-CAUB2 14 1 15804J

CAS Registry Numbers: 80-22-8 53-70-2 54-56-3 64-01-8 120-12-7 120-00-0 151-07-1 151-24-2 161-24-4 162-67-2 152-20-5 152-43-1 156-56-0 202-12-3 206-56-2 206-44-0 207-06-5 .analysis Identifiers: dein exhaust gases combustion emissions gas chromatog

216-01-9 236-64-6 243-17-4 27208-37-3 58615-36-4 72957-39-2

The Control of the

CA092100876017 92067601

Separation and identification of sulfur-containing polygolic areastic hydrocarbons (thiophers derivatives) from some PAM (polymolear areastic hydrocarbons) Author: Karchor, W.; Depaus, R.; Van Eijk, J.; Jacob, J. Location; Jt. Res. Cent., Comm. Eur. Communities, Petten,

Section: Cacecook Publ Class: COMF PROC Journal: Polymuci. Aromet. Hydrocarbons, Int. Symp. Chem. 1601. - Carcinog. Mustagen., 3rd Coden: 4185A. Publ: 79 Pages: 341-56. Meeting Date: 78 Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich

Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich Avail: Jones, Peter W.; Laber, Philip Identifiars: thiophene identification polycyclic aros hydrocarbon, polycyclic aros hydrocarbon analysis thiophene, pyrane analysis thiophene, benzopyrane analysis thiophene, benzopyrane analysis thiophene, thiophene, benzopyrane analysis thiophene, benzoperviene analysis

CA092 100876017

Descriptors: Arcestic hydrocarbons,polycyclic,anelysis Identifiers: thiophene deriv identification derivs benzoflucranthene pyrene benzoperylane benzopyrane CAS Registry Numbers: 80-32-8 110-02-10 129-00-0 191-24-2 194-68-0 206-82-3 242-53-8 30786-82-0 31473-75-3 72072-20-9

CA082 1008 150 IP

在有关的,这个时间,他们就是这个时间,我们就是这个时间,我们就是这个时间,我们就是这个时间,我们就是一个时间,我们就是一个时间,我们是这个时间,我们是这个一个时间 第一个时间,我们就是一个时间,我们就是一个时间,我们就是一个时间,我们就是一个时间,我们就是一个时间,我们就是一个时间,我们就是一个时间,我们就是一个时间,我们

Bevelopment of a protestype instrument for field sonitoring of polymolear aromatic hydrocartons (PAH) vapors
Author: Heuthorne, A. R.; Thorngate, J. H.; Gemege, R. B.; Location: Health Saf. Res. Div., Oak Ridge Natl. Lab., Oak Ridge, IN, 37830, USA Section: CAO58002, CAO73XXX, CAO60XXX Publ Class: COMF

Journal: Polymuci, Aromat, Hydrocarbone, Int. Symp. Chem. Bloil. - Carcinog, Mariagen., 3rd Coden: 41854. Publ: 79 Pages: 289-314. Meeting Bate: 78 Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich Address: Ann Arbor, Mich Address: Ann Arbor, Mich Interes: Philip Identifers: polymucies are an appointmental analysis.

CA092 1008 150 1F

Descriptors: Air analysis; Aromatic hydrocarbons, polynuclear-.emalysis; Spectrochamical analysis, UV; Spectromaters, UV Identiflers: biological studies second deriv UV spectra detn

CAS Registry Numbers: 85-01-8 91-20-3 120-12-7 129-00-0

8

Same analytical aspects of the quantitative determination of polymedia-armstics bydrocarbons in fugitive emissions from coal lights estimate processes.

Author: White, Curt M.; Sharkey, A. Q., Ur.; Lee, Milton L.; Location: Pilitaburgh Pa. 18212, USA

Location: Pilitaburgh Rengy Technol. Cent., Dep. Energy, Pilitaburgh, Pa. 18212, USA
Section: CA080002, CA081XIX, CA080XXX Publ Class: COMF

Journal: Polymuci. Aromat. Hydrocarbona. Int. Symp. Cbem. Biel. - Carcinog. Matagan., 3rd. Coden: 4195A. Publ: 79 Pages: 26:78. Meating Date: 78
Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich Avail: Jones. Peter W.; Laber, Philip Identifiers: polymuciaer arom hydrocarbon cost liquafaction, gas chromatog polymiciaer arom hydrocarbon. Tenax sampling polymiciaer hydrocarbon air

CA082 1008 1500M

Descriptors: Air analysis; Aromatic hydrocarbons, polynuclear-

Identifiers: dain liquefaction fraction gas chromatog mass operfreecopy sampling ambient apectrometry estations are selected. CAS Registry Numbers: 83-22-9 88-01-8 84-73-7 90-12-0 81-20-3 81-57-6 82-52-4 119-64-2 120-12-1 . analysis; Coal

Determination of polynuclear, aromatic hydrocarbons in the working environment
Author: Boorseah, Aif
Author: Boorseah, Aif
Location: Cant. Inst. Ind. Res., Daio, Morway
Section: CA05001, CA051XX, CA05XXX, CA080XXX Publ
Section: CA05001, CA051XXX, CA05XXX, CA080XXX Publ
Section: CA05001, CA051XXX, CA05XXX, CA080XXX Publ
Section: Cancing Mattingen, 3rd Coden: 4185AL Publ: 79
Publs Sall: Area secting Date: 78
Publisher: Ann Arbor Sect. Address: Ann Arbor, Mich
Avall: Uniona, Peter W.; Laber, Philip
Identifiers: Polynuclear arom Pydrocarbon air pollution, code plant polynuclear arom Pydrocarbon, aluminum plant polynuclear arom Pydrocarbon, aluminum plant hydrocarbon

CA092 1008 137 1W

Descriptors: Aromatic hydrocarbons, polynuclear, biological studies; Carbonization and Coking: Health hazard; Particle size;

Identifiers: air pollution aluminum coke plants relation working environments environment plant workers mutagenic activity

CAS Registry Numbers: 80-32-8 86-85-3 88-01-8 120-12-7 120-00-0 181-07-1 191-24-2 191-26-2 192-97-2 193-39-8 195-19-7 196-85-0 206-86-3-3 206-96-2 206-44-0 207-06-9 217-85-4 218-01-9 7429-90-5 26914-18-1 30777-18-5 30777-18-6 31711-13-2 41593-25-3 41593-26-4 56615-36-4 47) User 3631 2apr81 90 DIALOG Filed: CA Search 80-81/Vol 94(12) (See file 104) (Copr. An. Chem. Soc.) (Item

Sace 1370 Cadd2 1008 1370V

Section Cadd2 1008 1370V

In settled dust by Nigh-perference Hquid chromatography with mailti-length delection.

Muthor: Fechner, Detief; Selfert, Bernd
Location: Inst. Wasser., Boden- Lufthyg. BundaspaundhaitsaLocation: Inst. Wasser., Boden- Lufthyg. Bundaspaundhaitsamass. Berlin, Fed. Mep. Ger.
Section: CADSOO: CADSOXX Pub! Class: CDWF PRDC
JOURNAIL: Polymel. Armat. Hydrocarbona. Int. Symp. Chem.

Riol. - Carcinog. Natingen., 3rd Coden: 4185A. Publ: 79
Papas: 1819 Heating Date: 78
Papas: 181: Jenes. Peter W.; Leber, Philip
Identifiers: polymeclear arm Arbor dust, fluorescence
polymeclear arm hydrocarbon dust, fluorescence

CA082 1008 1370V

Descriptors: Air analysis;Air pollution;Aromatid hydrocarbona,polymuclear,analysis;Chromatography,column and liquid, high-performance;Dust;Spectrochamical analysis;fluoro-

CAS Registry Mumbers: 50-32-8 86-01-8 64-73-7 130-12-7 129-00-0 181-07-1 181-34-2 182-87-2 198-68-0 205-99-2 206-44:0 207-08-9 30777-19-6 Identifiars: detn fluorescence detector detection contg

CA082 1008 13698 120° 1368

identification of polynuclear areastic hydracarbon mix-urus in high-performance liquid chromolography fractions mix-urus the Spol'skil effect Author: Colesion, Anders; Stenberg, Ulf Location: Dep. Anders; Stenberg, Ulf

Publ Class: CONF Saction: CA058001, CA072XXX, CA080XXX Journal: Polymic! Aromat. Hydrocarbone, Int. Symp. Chem. Biol. - Careling, Mitagen., 3rd Coden; 41854, Publ; 79 Pages: 121-39 Meeting Date: 78 Publisher: Ann Arbor Sci. Address: Ann Arbor, Mich

Avail: Jones, Peter W.; Leber, Philip Edentifiers: polymuclear area hydrocarbon exhaust questilinear fluoroescence polymuclear area hydrocarbon

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Descriptors: Arcmatic hydrocarbona.polymuclear.analysis; Spectrochemical analysis,fluorometric dentifiers: dein exhant gases high performance liq chromatog quasilitates fluorescence chromatog quasilitates fluorescence chromatog quasilitates fluorescence chromatog quasilitates fluorescence chromatog analysis hambers: 80-22-8 86-86-3 128-00-0 191-07-1 191-24-2 191-26-4 192-87-2 207-08-9 2281-21-7 2363-12-8 CA082 1008 13698

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egomines caosinomines the phase system in the analysis of polymusian of the phase system in the analysis of polymusian system (PMA) from dissel engine andwarf by high performance liquid chromatography (PPLC) Author: Roumelloits, P.: Unger, K. K.; Tesarek, Q.: Authorise: Soumelloits, P.: Unger, K. K.; Tesarek, Q.: Location: Inst. Anorg. Chem. Anal. Chem., Johannes

Anal, Chem., Johannes Rep. Ger. Location: Inst. Anorg. Ches. Anal. Chem., Johan Gutenberg-Univ., Mainz, D-6500, Fed. Rep. Ger.
Section: GA05900! Publ Class: JOURNAL
JOURNAL: Fresenius' 2. Anal. Ches. Coden: ZACFAU Pu
19 Series: 298 Issue: 4 Pages: 241-9
Identifiers: diesel exhaust polynuclear arom hydrocarbon

CA092 1008 1365X

Descriptors: Arcestic hydrocarbons.polynuclesr.anslysis Identifiers: detn diesel exhaust phase system optimization

Portable fluoromatric monitor for detection of surface contamination by polymeisar aromatic compounds Author: Schuresko, Daniel D. Location: Chem. Technol. Div., Dak Ridge Natl. Lab., Oak Ridge, IN, 37830, USA. Section: CAGGIOSE, CAGGRAXX, CAGGOXXX. Pub! Class: JOURNAL JOURNAL Anal. Chem. CAGGIO: AMCHAM Pub!: 80 Series: 52 Issue: 2 Pages: 371-3

62 issue: 2 Pages: 371-3 Identifiers: safety area hydrocarbon fluorometry, industrial hygisies area hydrocarbon. cosl liquefaction hydrocarbon fluorometry. waste cosl liquefaction fluorometry.

CA092 10079253R

Descriptors: Aromatic hydrocarbons.analysis;Coal;Environment;fuel oil;Petroleum, synthetic;Shale oils;Solvents;Spectrochemical analysis;fluorometric;thates
Identifiers: dein industrial hygiene monitor liquefaction relation pollution monitoring fluorescence polynuclear aroms portable app

Matheds for characterization of complex mixtures of polymedisar arematic hydrocarbons.

Author: Snock, M. E.; Saverson, R. F.; Higman, H. C.; Arrendale, R. F.; Chortyk, D. Y.

Location: Job. Health Res. Lab., Sci. Educ. Adm., Athens. GA.

Publ: 79 Section: CAOSSODO, CAOSOXIX Publ Class: CONF PROC Journal: Polymuch. Arceat. Mydrocarbons, Int. Symb. 8101. - Carcings. Museum. 3rd Coden: 41834. Pub Peges: 231-60. Meeting Date: 78. Address: Ann Arbor Sci. Address: Ann Arbor Mich Avail: Jones, Pater W.; Laber, Philip. 1dentifians: raview cigars: saoke condensate and polycyclic arce hydrocarbon analysis review

condensate analysis,

Descriptors: Air analysis; Aromatic hydrocarbons, polycyclic, analysis; Tobecco smoke and smoking, condensates identifiers: characterization cigaret relation polymyclear CA08208063683V

9205:475 CA0020003:475R Characterization of environmental semples for polynuclear characterization by an x-ray excited optical luminescence technique

Author: Woo, C. S.; D'Silva, A. P.; Fassel, V. A. Gooli, USA Sociion: Dep. Chee., Town State Univ., Ames, IA, 60011, USA Sociion: CAOBOOGE, CAOSIXX, CAOSSIXX, Publicias: United Lass: United Coden: ANCIAM Publ: 80 Saries: Usurnal: Anal. Chee. Coden: ANCIAM Publ: 80 Saries: 1 Saries: 1 Pages: 189-64

Identifiers: polynciaer are hydrocarbon Identification environment: K ray excited luminescence hydrocarbon. Combustion analysis polynciae. area hydrocarbon. constraion coal analysis area hydrocarbon, conversion coal analysis area hydrocarbon, conversion coal analysis area hydrocarbon, conversion coal analysis. oil analysis arom hydrocarbon

Descriptors: Air analysis; Arcastic hydrocarbona, polytuclear, analysis; Ashes(rasidues), fly; Coal; Ervironaent; Fuel gas aenufacturing, gas; floation; Fuel oll; Shale olls; Solvents; Spectrochestal analysis, Luminacence, x-ray accited analysis, Luminacence, x-ray accited analysis, partied analysis, Luminacence, x-ray accited analysis, partied analysis, partied analysis, partied analysis, partied analysis, partied analysis, partied analysis and setting artied products at a processing CAS Registry Numbers: 80-32-8 83-70-3 86-49-5 86-55-3 86-00-0 132-65-0 189-56-9 189-64-0 191-24-1 191-24-2 192-97-2 184-56-2 196-56-0 206-44-0 217-59-4 218-01-5 CA09206061475R

Section: CADSIO2S, CADSOXXX Publ Class: JOURNAL JOURNAL JOURNAL: Fuel Coden: FUELAC Publ: 78 Series: S8 Lasue: 11 Peges: 783-9 Identifier: polyruclear area aliph coal, gas capillary Identifier: polyruclear area aliph coal, gas capillary from the Coal, mass spectrometry hydrocarbon coal, solvent refined coal assay, partition hydrocarbon coal solvent, cycle oil coal liquefaction. capillary gas chromatography/mass spectremetry
Author: Schultz, Rosemry V.; Jorgenson, James W.;
Mastarinec, Michael P.; Novotry, Milos; Todd, Lee J.
Location: Dep. Chem., Indiana Univ., Bloomington, IN, 47405,
USA

Descriptors: Alkanas, analysis, Aromatic hydrocarbons, polycyc-ifc, analysis; Circomatography, ges; Coal, solvent-refined; Mass Identifiers: detn capillary combined aliph CA09206044443A

92016451 CA09203016451H
Determination of polymuclear aromatic hydrocarbons in sediment by mass fragmentography
Author: Matsushims, Hajims; Hanya, Takahisa
Location: Fac., Sci., Tokyo Metropol. Univ., Tokyo, 158, Janan

nedel

P.cb : 79 ent Japan, polymuclear Coden: ABCHA6 Journal: Agric. Biol. Grem. Coden: ABCHA Serles: 43 Issue: 8 Pages: 1633-9 Identifiers: arom hydrocarbon dein sediment fragmentog arom hydrocarbon sediment, pol hydrocarbon sediment fragmentog Publ Class: JOURNAL Section: CA004001

Descriptors: Aromatic hydrocarbons.polynuclear,analysis; Chromatography,gas;Geological sediments;Mass spectroscopy identifiers: datn Japan fragmentog combined CAS Registry Numbers: 50-32-8 53-70-3 56-49-6 56-56-3 65-01-8 120-12-7 129-00-0 191-24-2 192-97-2 198-55-0 206-44-0 218-01-9 CA0920301645 IH

9204443 CA0920604443A
Characterization of polymunical promotic and aliphatic
hydrocarbon fractions of solvent-refined cost by glass

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Diatod Filed: CA Search &O-81/Vol 84(12) (See file 104) (Copf. Am. Chem. Soc.) (liem. 44 of. 47) User 3631 2spr81

92014900 CAD9202014900E
Use of micelles in the TLC separation of polynuclear
arometic compounds and emine acids
Author: Arestrong, Daniel W.; McNeely Narguerite
Location: Dep. Chem., Soudoin Coll., Srunswick, ME, 04011,

Section: CAOSCOO4 Publ Cless: JOURNAL
JOURNEL: Anal. Lett. Coden: ANALSP Publ: 79 Series:
Identifiers: thin layer chromatog sicelle reagent, amino
ecid thin layer chromatog, and hydrocarbon thin layer
chromatog, polymucleer area thin layer chromatog,
dodecylasifate solvent thin layer chromatog, activity layer
excent thin layer chromatog, octylaulfosuccinate solvent thin layer chromatog.

CA09202014900E

Peacriptors: Amino acida, analysis; Aromatic hydrocarbons, polymician, analysis; Chromatography, thin-layar; Micalias il danititiates: micaliar agolium dodecylsuifate soins eluent CAS Registry Numbers: 80-32-8 81-36-4 82-90-4 66-40-6 86-85-9 86-86-0 172-18-4 81-96-8 81-36-4 83-90-8 81-36-4 83-90-9 173-32-5 14-79-3 85-01-8 86-73-7 91-20-3 120-12-7 129-00-0 147-88-3 181-21-3 182-87-2 188-58-0 577-11-7 8867-09-9

CA09202010606R 92010606

Factor analysis and derivation of an experimental equation on polymolear areastic hydrocarbon asissions from automobiles Author: lands, Takahii yamamira, Takahii Kato, Yoshihiro; Location: Fac. Sci., Sci. Mniv., Tokyo, Tokyo, 162, Japan Setion: G.069002 Publ Class: UDURNAL Coden: ESTHAG Publ: 79 Setion: Eviron. Sci. Technol. Goden: ESTHAG Publ: 79 Setion: distain 19 Issue: 9 Pages: 1077-81 Identifiers: automobile amission polycyclic arom hydrocarbon exhaust polycyclic arom hydrocarbon equation

CA09202010606R

Descriptors: Aromatic hydrocarbons.polycyclic,uses and sizeslangous.Exhaust gases identifiers: automobile calon equalion mileage angine officeletton emission caloin.

CAS Registry Numbers: 50-32-8.

S2010470 CA082020104705
Batershnatten of average esission rates of polynuclear ramatic plyshocarbons from a sattemobile
Author: Hende, Takeshi; Venemura, Takeki; Kato, Yoshiro; Salto, Sholchiro; Ishii, Jadahiro
Location: Fac. Sci., Sci. Univ. Tokyo, Johan
Section: CA086001 Fubi Class: UDURNAL

Publ: 79 Journal: Talki Dsen Gakkajshi Coden: 705GDC Publ: 77 Series: 14 Issue: 3 Pages: 98-105 Language: Japan Identifiers: polynuclear arom hydrocarbon exhaust sampling

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CA092020104705

Aromatic hydrocarbons, polycyclic, analysis; Descriptors: Aromatic hydrocarbons, poly Exhaust gases: Sampling Identifiars: detn comparison methods method

Part I: The synthesis and cheracterization of some modernor carboranes and derivatives. Part 2: Characterization of the polymollar around hydrocarbon and alignatic fractions of the polymollar around hydrocarbon and alignatic fractions of colvent refined coal by Author: Schultz: Researy V. Location: Schultz: Researy V. Location: Indiana Univ. Bloomington, IN. USA Section: CA026001, CA023XXX, CA028XXX, CA051XXX Publ

Section: CA026001, CA023XXX, CA028XXX, CA051XXX Publ Class: DISS
Coden: DASSEA Publ: 79 Pages: 118 pp.
Citation: Diss. Abstr. Int. 8 1979, 40(2), 744
Avall: Univ. Microfilms Int., Order No. 79:6925
Identifiers: Carborane monocarbon, hydrocarbon characterization solvent refined coal, arom hydrocarbon polynyclear coal

CA09201006293U

Descriptors: Aromatic hydrocarbons,polynuclear,properties; Carboranes,monocarbon;Coal,solvant-refined; Hydrocarbons,allph.,properties Identifiers: characterization prepn

1 of 87) User 3631 2apr81 Print 13/2/1-87 Dialog File104: CA Search - 77-79/Vol 91(26) (Copr. Am. Chem. Soc.) (Item

CA09126222066U

Repid anniysis for polynuclear aromatic hydrocarbons by innear-susep differential pulse voltametry.

Innear Surrows, Kerlyn C.: Hughes, Michael C.

Location: Dep. Chem., Lenigh Univ., Bathlehem, PA., 19015.

Publ: 79 Saction: Cacacoco Publ Class: JOURNAL ACACAM Publ: 73 Journal: Anal. Chia. Acta Coden: ACACAM Publ: 73 Series: 110 lasus: 2 Pages: 255-60 Identifiers: polynuclear arom hydrocarbon detn voltammetry

91205152 CA091252061528
Rapid simple sample preparation technique for analyzing Rapid simple sample preparations to sediments by gas chromatography—ass specificantry
Author: Tan, Vulin L.

Location: Environ. Meas. Lab., Dep. Energy, Mey York, MY, 10014, USA.
Section: CA004001 Publ Class: UDRBAL
Section: CA004001 Publ Class: UDRBAL
Section: CA004001 Publ: 79
Series: 176 Issue: 3 Pages: 319-27
Identifiers: aros hydrocarbon sediment chrostog spectrometry, polynuclear aros hydrocarbon detn sediment, gas operando aros hydrocarbon sediment, sass spectrometry aros hydrocarbon sediment.

91203898 CA09124203898A K-ray excited optical luminascence of polynuclear arcmatto Mydrocarbons Author: Destraich, G. J. Location: Assailab., Assailab. USA
Section: CAOMODOS Publ Class: YECH REP
Journal: Report Coden: DOMEP3 Publ: 79 Issue
15-T-856, Pages: 164 pp.
Citation: Energy Res. Abstr. 1979, 4(16), Abstr. No. 43u03

analysts Identifiers: polynuclear arom hydrocarbon luminescence, x ray excited luminescence analysis Analysis of Raritan Bay bettom waters for polynuclear Analysis of Raritan Bay bettom waters for polynuclear accentic hydrocarbons Author: Stainken, Dennis; Frank, Use Location: Ind. Environ. Res. Lab., EPA, Edison, MJ, 08817,

Section: CAOS-1001 Publ Class: JOURNAL Coden: BECTAS JOURNAL Sells. Environ. Contem. Toxicol. Coden: BECTAS Publ: 79 Seles: 22 Issue: 4-6 Pages: 480-7 Identifiers: polynuclear area hydrocarbon Raritan Bay.

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arom hydrocarbon, fluorescence spectroscopy polymiclear chromatog liq polymicles arom hydrocarbon

2

Identifiers: phosphorimetry polynuclear area detn, fossil fuel polynuclear area, coal liquefled phosphorimetry area, synthetic petroleum phosphorimetry area. I STOLE: Rapid analysis of PMA (polynuclear aromatic) compounds complex samples by room temperature phosphorimetry Author: Vo-Dinh, T. Location: Oak Ridge Nati. Lab., Dak Ridge, TN. USA Section: Cack Ridge Nati. Lab., Dak Ridge, TN. USA Section: Cack Ridge, Coden: Dake Paper: FCH REP Ournal: Report Coden: DakeP3 Publ: 76 Issue COMF-781150-1, Pages: 11 pp. 4(9), Abstr. No. 24065 Avail: NIS

Separation and identification of polynuclear aromatic compounds in coal tar by using glass capillary chromatography including combined gas-Chromatography-mass spectrometry. Author: Borwitzky H.; Schomburg, G. Location: Max-Planck-Inst. Mohlenforsch., Muelheim/Ruhr, D-430, Fed. Rep. Ger. Section: CAGGIOZS, CAOGOXX Publ Class: JOURNAL Journal: J. Chromatogr. Coden: JOCRAM Publ: 79 Series: 170 Issue: i Pages: 99-124 Identifiers: coal 'ar polynuclear arow, chromatog hydrocarbon coal tar, gas chromatog coal tar, mass spectroacopy coal tar, mass 91177782 CA09122177782M

Analysis of polymusiser arematic hydrocarbons Author: Brown, Raiph A.; Searl, Thomas D. Location: Anal. Inf. Div., Exxon Res. and Eng. Co., Linden, 91177571 CAO9122177571S

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MJ. USA Setion: GAOSIODO, CAOSOXXX. Publ Class: JOURNAL Setion: Chromatogr. Sci. Coden: CHGSAL Publ: 79 Series: 11 Issue: Chromatogr. Pet. Anal. Pages: 367-94 Identifiers: review polymicleer arom hydrocarbon analysis: gas chromatog arom hydrocarbon review. UV spectroscopy arom hydrocarbon review.

87) User 3631 2apr81 • DIALOG Filei04: CA Search - 77-79/Vol 91(26) (Copr. Am. Chem. Soc.) (Item

CADDIZOIG7966K
excited optical luminescence of polynuclear arcmatic

analysis Author: Gestraich, Gregory Joseph
Location: Comescient John State Univ., Ames. 14, USA
Section: CAGGOOG Publ Class: DISS
Coden: DASSEA Publ: 79 Pages: 16: pp.
Citation: Diss. Abstr. Int. 8 1879, 40(1), 203
Avail: Univ. Microfiles Int., Order No. 79:620s
Identifiers: polymaciaer aros hydrocarbon
Luminescence, x ray axcited luminescence hydrocarbon Polymorlear arosatic hydrocarbons associated with coal combustion.

Author: Sucre, L.; Jennings, W.; Fisher, G. L.; Raabe, D. G.; Discino, J.; Cartion. Dec., Location. Dec., Discino, J.; Castion. Dec., Discino, J.; Castion. Cade Sci. Technol., Univ. California, Davis, Section. Cade Soc. Publ. 10. S.; Coden: XMESAV Publ. Jess: J. Coden: XMESAV Publ. The Series: 519 Issue: Trace Org. Anal.: New Front. Anal. Ches. Pages: 109-20. Identifiars: coal combustion polynuclear arom hydrocarbon.

Biid5344 CAOBiimid53440
Bevelopment of a prototype instrument for field monitoring of PMA (pelynacian prompting compound) vapors
Author: Hauthorne, A. R.; Thorngate, J. H.; Gesmage, R. B.; Location: Oak Ridge Matt. Lab., Oak Ridge, IN, USA Section: CAOSSOO3, CAOSIXXX, CAOSXXX Publ Class: YECH Journal: Report Coden: 038EP3 Publ: 78 Issu CMF-781038-1, Pages: 21 pp. Citation: Energy Res. Abstr. 1979, 4(6), Abstr. No. 14147 Avail: MIS Identifiers: polymuclear arom compd detn spectrometer 91145287 CADSISSISSIN Preliminary thoughts on proxy PNA (polynuclear aromatic) Compounds in the vaper and solid phase Author: Games, R. S. Author: Games, R. S. Location: Games, R. S. Section: Cadesoca, R. S. Section: Cadesoca, Cadesoca, Cadesoca, IN, USA Section: Cadesoca, Cadesoca, IN, USA Section: Cadesoca, Cadesoca, IN, USA Class: IECH RE JOURNALL Report Coden: DOMEP3 Publ: 78 Issue: Citation: Energy Res. Abstr. 1979, 4(8), Abstr. No. 21288 Avail: NIS

geta. Identifiers: polynuclear area hydrocarbon proxy environment polynuclear area hydrocarbon proxy

9

91145161 CAO9118145161S
On the analytical potential of micro-Raman spectroscopy in the trace characterization of polynuclear arcmatic DC, 30234, USA
Section: CA08001, CA081XX, CA080XXX Publ Class: UDURNAL
Journal: NES Spec. Publ. (U. S.) Coden: XMSSAV Publ:
Udurnal: NES Spec. Publ. (U. S.) Coden: XMSSAV Publ:
TO Saries: 619 Issue: Trace Org. Anal.: New Front.
Anal. Chem. Pages: 723-9
Identifiers: arom hydrocarbon detn environment spectroscopy d. Location: Cent. Anal. Chem., Natl. Bur. Stand., Washington, Kurt f. hydrocarbons Author: Etz, Edgar S.; Wise, Stephen A.; Heinrich,

A Comparison of some chromatographic methods for the estimation of some chromatographic methods for the estimation of polynuclear aromatic hydrocarbons in pollutants Author: Gurchill, P.; Herod, A. A.; Usees, R. G. Location: Coal Res. Establ., Natl. Coal Board, Chaitenhan/Glos., Engl. Section: GAOSOOI, GAOSOXX Publ Clear; TECH REP Journal: Comm. Eur. Communities, (Rep.) EUR Coden: CECED9 Pournal: Comm. Eur. Communities, (Rep.) EUR Coden: CECED9 Palorization Coal, Pages: 206-23 Meeting Date: Ty Identifiers: polymuclear arom hydrocarbuna meth. chromatog benzopyrene coke oven

1 Author; Frycks, Josef Loss Tar Chem., Urxovy zevody N. P., Valesske Merfrici, 787 27, Csech.
Section: Gobool, CadBixXX Publ Cleas: JOURHAL Journal: J. Chromatogr. Coden: JOCRAM Publ: 79 Section: Gobor: JOCRAM Publ: 79 Section: Gobor: JOCRAM Publ: 79 Section: Passes: 2 Pages: 488-9
Identifiers: polyvuclear arom hydrocarbon sid contemination, gas chromatog polyvuclear arom sid, phenanthrene sid contamination. 91101542 CAO9112101542N Contamination of some polymician aromatic standards

Signator chosisosatora Bautisa liquid chromatographic method for assessing polymusians areastic hydrocarbon pollution in fresh valor avairements
Author: Black, J. J.; Dymerski, P. P.; Zapisak, M. F. Location: Roseril Park New. Inst., Div. New York State Dap. Location: Moseril Park New. Inst., Div. New York State Dap. Location: Cods.002, USA. Section: Cods.002, CADTSXXX. Publ Class: JOURNAL Journal: Bull. Environ. Contam. Porticol. Coden: RECIAS Publ: 79 Series: 22 Issae: 1-2 Pages: 278-84 Identifiers: polymuclear area hydrocarbon ests river

Stodesde Cade: 10002566E

Bevolegeant of an equeue pelynuclear aromatic hydrocarban standard redermon amicalia;
Author: May, W. E.; Broun, J. M.; Chesler, S. M.; Guenther, F.; Hilpert, L. R.; Hertz, H. S.; Wise, S. A.

Location: Matt. Bur. Stand., Weshington, DC, 20234, USA
Section: Code0003 Publ. Class. UDIMMA.

Journel: MS Spec. Publ. (10. S.) Coden: XMBSAV Publ.;

Andl. Chem. Bit leave: Trace Org. Anal.: New Front.
Anal. Chem. Pages: 219-24
Identifiers: sid red polynuclear arom hydrocarbon, 11q
chromatog polynuclear arom hydrocarbon.

Stoking Cadelogosing Vermination of polynuclear aromatic forematographic determination of polynuclear aromatic foreignment in the environment of the cation: toroid, a. M.; Lysyuk, L. S.; Location: Inst. Phys. Chem., Miev, USSR; JOHNAL, Chem., Coden: ZANGAR, Publ: 79 Series: 34 Issue: 3 Pages: 877-80 Language: Russ Identifiers: review polynuclear arom hydrocarbon detn, gas chromatog arom hydrocarbon review

9:002424 CA09:040024340
Application of second-derivative UV absorption spectromatry to polywedness escend-derivative UV absorption spectromatry to polywedness escention and polymers, and the second analysis and Authoris Habits at Alam B.: Thorngate, John H. Location: Habits at Alam B.: Thorngate, John H. Lab., Oak Ridge, IN, 37830, USA.
Section: CA060000, CA068XX Publ Cleas: JOHNHAL Journal: Appl. Section: Caden: Appl. 79
Section: 23 Issue: 2 Pages: 20-5
Identifiers: UV second deriv polymelear area dein UV photometry, health protection polymelear area

Sibilish CADSID2013188V Enformance liquid chromatographic separations of polymolear aromatic hydrocarbons. Temperature as a separation parameter Author: Christian parameter Author: Christian Canada Cent. Miner. Energy Technol., Ottawa, ON, KiA Odi, Can.
Section: Canada Cent. Miner. Energy Technol., Ottawa, ON, KiA Odi, Can.
Section: Canada Cent. Miner. Energy Technol., Ottawa, ON, Section: Canada Cent. Miner. Energy Technol., Ottawa, ON, Section: Concestogr. Sect. Cadan: JOHSEZ Publ: 79 Series: 17 Issue: 8 Pages: 248-52 Identifiers: entropy high performance itq chromatog, polymuciaen erom hydrocarbon liq chromatog, temp effect liq chromatog thermoch

Bioceste caceioscoested of polytuclear aromatic hydrocarbon determination in water Author: Budkiewicz, T.; Ryborz, S.; Maslowski, J. Location: Cent. Environ. Dev., Res. Inst. Erviron. Dev., Katowice, Pol. Cont. Environ. Dev., Katowice, Pol. Colexion: Caceiooz, Cacitoxx, Cacesxxx, Caceoxxx Publ class: Journal: Erviron. Prot. Eng. Coden: EPENDE Publ: 78 Series: A Issue: 3 Pages: 283-6 Identifiers: polynuclear arom hydrocarbon detn water, air polynuclear arom hydrocarbon detn

Analysis for polynuclear areastic hydrocarbons in working atmospheres by computarized gas chromatography-mass spectromatry
Author: Bjoerseth, Alf; Etlund, Goran
Location: Cent. Inst. Ind. Res., Oslo, 3, Norway
Section: Cadesoo2, Codoxxxx Publ. Class., UDURNAL
Journal: Anal. Chim. Acta Codoxxx Publ: 78
Series: 106 Issue: 1 Pages: 119-28
Identifiers: polynuclear arom hydrocarbon detn air

87) User 3631 2apr81 22 of DIALOG FILE104: CA Search - 77-79/301 94(26) (Copr. Am. Chem. Soc.) (Item

sionasts caosiosopasts The separation and determination of polymusical aromatic Mydrocarbone by high-performance liquid chromatography: Part

British Carbonization Research Assoc.
Location: Chesterfield/Derbyshire, \$42 &45. Engl.,
Location: CA088002, CA081XX Publ Cless: JOURHAL
JOURNEL: Carbonization Res. Rep. Coden: CRREDS Publ:
76 Series: 28, Pages: 19 pp.
Identifiers: arom hydrocarbon data fuel gas, benzopyrene

dein chronetog fuel ges

in shellfish by

E SRRDS

chroms tog

Montysis of polymolear aromatic hydrocarbons using high-speed liquid chromategraphy Author; Kamaia, Kunihiro; Kan, Teruo; Vamazce, Ritsuko; Herada, Hirofual 15

Section: Co00004 Publ Class: JOURNAL denkyu Nempo Journal: Tokyo-toritsu Eisei Kenkyusho Kenkyu Nempo John: Tother: Teles: 28-1, Pages: 95-9 Language: Japan Language: Japan John Journal Journal Journal Journal Journal Journal Journal of Chromatog, polymolear area hydrocarbon liq chromatog, benzpyrene liq chromatog Location: Tokyo Metrop. Res. Lab. Put'ic Health, Tokyo.

SoleSoli cassolistoito
Polynusiaar aremaila hydracarbane in food additives. (III).
Analysis of benzelalpyrane in caremai
Author: Hirokado, Masako; Makajime, Iuao; Usami, Hiroyuki;
Endo, fusayoshi
Location: Tokyo Metrop. Res. Lab. Public Health, Tokyo,

Japan
Section: CA01700: Publ Cless: JOLDMAL
Section: CA01700: Publ Cless: JOLDMAL
Coden: TREMAF Publ: 77 Sectee: 28-1,
Language: Japan
Identifiers: benzopyrene caramel detn

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solstone cansolsastoned between the food additives. (IV). Pelynales arcestic hydrocarbon in food additives. (IV). Analysis of benzo(1)pyrene in addivated carbon and carbon black.

Author: Nakajima, Iwao: Hirokado, Masako; Usami, Hiroyuki; Mizoiri, Shigaru; Endo, Fusayoshi Location: Tokyo Metrop. Res. Lab. Public Health, Tokyo, Japan

Saction: CA017001 Publ Class: JONRAL Journal: Tokyo-toritau Eiset Kankyusho Kankyu Nempo Codan: TRENAF Publ: 78 Saries: 29-1, Pages: 203-5 Language: Japan Japan Bannzopyrene detn carbon black, activated carbon bennzopyrene detn

CA090221737952 90173795

Determination of benzo(a)pyrene and other polynuclear areation between the security of the second of chromation perticulate eaterial by ultrasonic extraction and reverse phase high pressure liquid chromatography for sawicki, E. Location; Golden, C.; Sawicki, E. Location; Off. Res. Dev., EPA, Research Triangle Park, N. C. Section; CA069002, CA060XXX Publ Cleas; JUCKNAL JOURNAL Locine; Anal. Lett. Coden; AMALBP Publ: 78 Series: Jucknal; 62 Pages; 1051-62

hydrocarbon detn air

90146358 CAGGOIS146368R POlymolear aromatic hydrocarbons Separated by thin-layer chromatography for detection with Shpol'sil [Sur-desperature Fluorescence Author; Colmsjo, Anders; Sterberg, Ulf Location; Dep. Ansl. Chem.

Section: CAOSOOO4 Publ Cleas: JOURNAL
Journal: J. Chromatogr. Coden: JOCRAM Publ: 79
Section: 169. Pages: 206-16.
Jentifiers: vacuum sublimation polynuclear arom hydrocarbon
thin layer chromatog arom hydrocarbon, fluorescence
detection polynuclear arom hydrocarbon, benzopyrene chromatog
sublimation fluorescence detection, person chromatog
sublimation fluorescence detection, benzoperylene chromatog
sublimation fluorescence detection.

Moltass Cabbolitasso Malysis of pelymician prematic hydrocarbone by glass capillary gas directory using simultaneous flass feeting and electron capture defection Author: Sjorseth, Alf. Ethund, Soran Author: Sjorseth, Alf. Ethund, Soran Costion: Cant. Inst. Inst. Sol. Moray Section: Cabbold, Cabbolt, Publ. Class: Uniquel Journal: HEC CC, J. High Resolut. Chromatogr. Chromatogr. Commun. Coden: HCJCSB Publ: 79 Series: 2 Issue:

Identifiers: polynuclear area hydrocarbon detn environment, gas chromatog polynuclear area hydrocarbon, environment analysis polynuclear area hydrocarbon, flase identation detection polynuclear area, electron capture detection polynuclear area.

90141591 CA08018141591P

Synchronous specifrecopy for analysis of polynuclear areastic compounds Author: Vo-Dinh, Tuen; Gammage, Richard B.; Hawthorne, Alan

R.: Thorngate, John H. Location: Health Saf. Res. Div., Oak Ridge Netl. Lab., Oak

P.C. 78 Bidge, Tern.
Section. CA065002 Publ Class: JOHNAL
Journal: Environ. Sci. Technol. Coden: ESTRAG
Series: 12 Issue: 12 Pages: 1297-302
Identifiars: polynuclear area compd dein water

Mitragen analogs of polynuclear aromatic hydrocarbons in tabecos sants
Author: Stock, N. E.
Location: Tob. Health Res. Lab., ABS, Athens, Ga.
Section: CADOA013 Publ Class: JOHANAL
JOHNNIE CACINGS. - Compr. Surv. Coden: CCSUDL Publ: 78 Series: 3 Issue: Polynucl. Aromat. Hydrocarbons Pages: 203-16
Identifiers: secte hetero analog polynuclear arom hydrocarbon, nitrogen analog polycelic arom hydrocarbon smoke

Mode tamperature phesphorescence of several phermacautical properations and polymodian aromatic hydrocarbons Author: Goues, Esther Alaire Location: Goues, Esther Alaire Location: Univ. Florida, Gainsaville, Fla. Section: CAOGOOS, CAOJAXX Publ. Class: DISS Coden: CAOGOOS, CAOJAXX Pages: 186 pp. Citation: Diss. Abat. 181. B 1879, 28(1), 3348 Avall: Univ. Microfilms Int., Order No. 7900006 Identifiers: phesphorimatry phermacautical arom hydrocarbon

analvate.. pharmaceutical analysts room temp phosphorimetry. Polynucinar arom hydrocarbon analysts phosphorimetry

Proceded CADD0130080040

The effect of sample environment on the room-temperature prosphereacons of saveral pelymiciaer encometic hydrocarbons Author: Boser, Esther Lus-Yen; Minefordner, J. D. Location: Dap. Chem., Univ. Florida, Gaineavilla, Fla. Saction: CADD400: Publ Class: UDIRMAL doubler Anal. Chim. Acta Coden: ACCAM Publ: 78 Series: 102, Pages: 1-13 Identifiers: arom hydrocarbon phosphorescence, tellurium efom hydrocarbon phosphorescence

CA09011085380E

High pressure liquid chromatographic determination of polyhidolear aromatic hydrocarbons in eysters
Author: House, James P.; Guerrero, Hamberto; Biehl, Edward
E.; Kenner, Charles T.
Location: Chol Drug Adm., Dallas, Tex.
Section: CA017001, CA004XXX Publ Class: UDWRAL
Journal: J. Assoc. Off. Anal. Chem. Coden: JANGA2
Publ: 79 Series: 62 Issue: f Pages: 29-35
Identifiers: polynuclear arom hydrocarbon oyster, ite

90075817 CAODO10075817P

Gel elution of haterocyclic enalogs of polynuclear arcmatic hydracarbons from bio-beads
Author: Snock, M. E.
Location: Tab. Lab., ARS, Athans, Ge.
Section: CAOSO02, CAOSOXXX, Publ. Cleas: JOHRHAL, JOHNHAL, JOHNHAL, Anal. Chie. Acta Coden: ACACAM Publ: 78
Series: 99 Issue: 2 Pages: 299-304
Identifiers: aros hydrocarbon detn sir chromatog, bio bead

gel elution

87) User 3631 2apr81 DiALOG FiletOt: CA Search - 77-79/Vol 91(26) (Copr. Am. Chem. Soc.) (Item 36 of

90043306 CA09006043308H Sanit Sanit Sanit Station of pelynaclear aromatic hydrocarbons by Sapol'stil low temperature (luarescence Author: Colmajo, Andere; Stemberg, Ulf Location: Dep. Anal. Chem., Univ. Stockholm, Stockholm,

section: CAOBBOO2, CAOTBUXX Publ Class: JOURNAL Journal: Anal. Chem. Coden: ANCHAM Publ: 79 Series: St. Issue: t Peges: 145-50 Identifiers: polynuclear arom hydrocarbon data fluorescence, exhaust polynuclear arom hydrocarbon detn, waste gas polynuclear arom hydrocarbon detn, waste gas

90039:16 CA09005039:16A Betweet Compounds, part III. Betermination of bifunctional compounds, part III. Popuration are selective fluorescent respents for HFILC and HFLC. Singhawangcha, S.; Zlatkie, A.; Author: Poole, C. F.; Singhawangcha, S.; Zlatkie, A.; Poole, C. F.; Singhawangcha, S.; Zlatkis, A.;

Morgan, E. D.
Localion: Dep. Cham., Univ. Houston, Houston, Tex.
Section: CA033006, CA023XXX, CA024XXX, CA029XXX, Publ Class: JOHNAL Journal: HRC CC, J. High Resolut. Chromatogr. Commun. Coden: MCJCDB Publ: 78 Series: 1

Identifiers: high pressure liq chromatog, thin layer chromatog bigh pressure, naphthelemeboronate pinacol chromatog echyston chromatog sepn, acdysterona phenanthranaboronate chromatog sepn, acdysterona phenanthranaboronate chromatog sepn. Chromatogr.

Modesta Cabboobsoasaa Multi-alkylated polymusiaar aromatis hydrosarbona of tobacco amede: asperation and identification Author: Snook, W. E.; Saverson, R. F.; Arrendale, R. F.; Hygman, H. C.; Chortyk, D. T. G. G.c. Adm., Athena, Ga. Location: Tob. Lab., SGI. Edg., Adm., Athena, Ga. Section: CAD11007, CAD098XX Publ Class: JOURNAL Journal: Beltr, Tabekforsch. Coden: BETAN Publ: 78 Series: 0 issue: 4 Pages: 222-47

Schools of the performance liquid chromatography of Separation by high performance liquid chromatography of pelynuclear arcentic Mydrocarbons listed by the World Health Ergenization as pellution indicators in drinking water Author: Munt, B. C.; Wild, P. J.; Crosby, N. T. Location: Dept. 14th, 14th, 24th, 24

chross top 12 fasue: 6 Pages: 643-4 Identifiers: phthelimidopropyltrichlorosilana packing, pojynucian arom hydrocarbon sepn

8

89220046 CAO8926220046X Officers analysis of polynuclear aromatic hydrocarbons - quantitative distribution in air of Ontario cities

Section: CA055002, CA079XXX Publ Class: JOURNAL JOurnal: Environ, Sci. Technol. Coden: ESTHAG Publ: 78 Series: 12 Issue: 8 Pages: 909-16 Identifiers: polycyclic arom hydrocarbon detn particulate, thin layer chromatog arom hydrocarbon, fluorescence apectroscopy polycyclic arom hydrocarbon, fluorescence Author: Katz, Moris: Sakuma, Takao; Ho, Andraw Location: Cent. Res. Environ. Qual., York Univ., Toronto,

A comparison of some chrometegraphic methods for estimation of polymiciaes aromatic hydrocarbons in pollutants
Author: Burchill P.: Herod, A. A.: James, R. G.
Location: Ceal Res. Establ., Nati. Con! Board,
Cholischan (Glos., Engl.)
Section: CAOSBOOL, CAOSOXX Publ Class: JOURNAL
JOURNAL GAOSBOOL, CAOSOXX Publ Class: JOURNAL
JOURNAL GAOSBOOL, CAOSOX Surv. Coden: CCSUDL Publ:
Section: 3 Issue: Polymuci. Aromat. Hydrocarbons

Identifiers: polynuclear area hydrocarbon analysis, gas chromatog polynuclear area hydrocarbon, mass spectroscopy polynuclear area hydrocarbon

89203113

9203113 CA089242031132 Analytical methods for polymiclear arematic hydrocarbona Author: Schmeltz, Irwin; Brunnemann, Klaus D.; Hoffmann, Dietrich

Location: Maylor Dana Inst. Dis. Prev., Am. Health Found., Mahlalia, N. Y. Section: GOODOO, CADBOXX Publ Class: COWF PRDC Journal: Prev. Datect. Cancar. (Proc. int. Symp.), 3rd Coden: 27ALAD Publ: 78 Series: i Issue: 2 Pages: 1973-92 Meeting Date: 74 Address: New York, N. Y

Identifiers: review polymolesr arom hydrocarbon datn, tobacco smoke arom hydrocarbon review Avail: Misburgs, Herbert E Identifiers: review poly

88199812 CAG8824189812Q A review of methods for the estimation of polynuclear prematic hydrocarbons with perticular reference to coke oven

Author: Merod, A. A.; James, B. G.
Location: Cost Res. Establ., Matt. Cost Board, Stoke Cocation: Cost Res. Establ., Matt. Cost Board, Stoke Orchard/Choitenhea, Engl.
Section: Ca068400, Ca0684XX, CA060XXX Publ Class: Jouenat Journal: Jinst. Fuel Coden: Jiflud Publ: 78
Series: Si Issue: 408 Pegas: 164-77
Identifiers: review polymerlear area hydrocarbon dath, coke oven emission hydrocarbon review, gas chromatog area hydrocarbon review, and hydrocarbon review, and hydrocarbon review, area hydrocarbon review, and hydrocarbon review, and hydrocarbon review.

Estatis CADSSOISSIES POLYMETER Promette hydrocarbons in Baterninstien of polymeter eromette hydrocarbons in Belly(viny) chiencies section particulates by high pressure Newisternessies prometers by the pressure Newisternessies appearamently Author: Liso, uchn C.: Browner, Richard R. Lachnol, Atlanta Location: Sch. Aerosp. Eng., Georgia Inst. Technol., Atlanta Section: CAGGGOO2, CAGGGAXX, CAGTGAXX, Publ Class: JOURNAL JOURNES: Anal. Chem. Coden: AMCHAM Publ: 78 Series: 80 Issue: 12 Pegas: 1821-6
Identifices: polynuclear arangement processing PVC secks chrossing PVC secks analysis

CA089 19 156222W

Analysis of palymentaer areastic hydrocarbons and erganochierins pallutants in Great Lakes herring gulls by high resolution gas chromategraphy.
Author: Hallett, Douglas J.; Moratrom, Ross J.; Gruska, Francis I.; Cosba, Michael E.
Location: Gan. Wildl. Serv., Dep. Fish. Environ., Ottawa, Publ Class: CONF PROC Section: CA004001

Glass Capillary Chromatogr., Int. Symp., 2nd AA Publ: 77 Pages: 116-28 : Hett. Chromatogr. Address: Ead Duerkhelm, Ger

Designate CACCESSES ACCESSES ACCESSES ACCESSES IN value. In Telegraphics of polymostar accesses to value. It is fittingles and recovery of aix representative compounds with polymosthere feams.

Author: Basu, Dipak K.; Saxens, Jitendra
Location: Cent. Chem. Hazard Assessment, Syracuse Res. Corp.,
Syracuse, M. Y.
Sections: M. Y.
Sections: M. Y.
Sections: M. Y.
Sections: Co. CAGBOXIX Publ Class: JOURNAL
JOURNAL: Environ. Sci. Fechnol. Coden: ESTIMG Publ: 78
Series: 12 Issue: 7 Pages: 791:5
Identifiers: aros hydrocarbon polymuclear detn water,
polyurathans aros hydrocarbon polymuclear detn water
aros polyurates.

89182407 CAO8918182407P Methods for polymiclesr arematic hydrocarbon analysis in the marine environment

Author: Wise, S. A.; Chesler, S. N.; Hariz, H. S.; Hilpert, L. R.; May, W. E. tocation: Inst. Mater. Res., Matl. Bur. Stand., Washington, D. C.

Section: CAGGOOX, CAGGOXX Publ Class: JOURNAL Journal: Carcinog. - Compr. Surv. Coden: CCSUDL Publ: Pages: 176-62
Pages: 176-62
Identifiers: hydrocarbon arom polynuclear dein seawater, sediment polynuclear arom hydrocarbon dein

89151729 CAC6918151729H

Measurement of polymodler arceatic hydrocarbons in disselublastigness
Author: Existence, B. A.: Spindt, R. S.
Author: Gulf Res. and Dev. Co., Pittsburgh, Pa.
Section: CAOSOO2 Publ Class: UDURAL
JOURNAL: S.K. (Tech. Pap.) Coden: SAEPCT Publ: 78
Series: 780115, Pages: 12 pp.
Identifiers: polymiclear arcm hydrocarbon exhaust, exhaust
gas hydrocarbon asseurement, dissel engine exhaust hydrocarbon
benzopyrene dissel engine exhaust, benzoenthracene dissel

CA08916135458P 89135458

Polymiclas somatic hydrocarbons in selected U.S. drinking waters and thair raw water sources
Author: Basu, Dipak K.; Saxens, Jitendra
Location: Cent. Chem. Hazard Assessment, Syracuse Res. Corp., Syracuse, N. V.
Section: CAOSIOD: Publ Class: UNUMMAL
Journal: Environ. Sci. Technol. Coden: ESTING Publ: 78
Section: 12 Issue: 7 Pages: 785-8
Identificats: arcom hydrocarbon polymiclas vater, drinking water polymiclas arce hydrocarbon

Heithed development and monitoring of polynuclear aromatic hydrocarbons in selected US taters
Author: Savens, U: Beau, D. K.; Kozuchouski, U. Location: Syracuse, N. Y. Saction: Syracuse Res. Corp., Syracuse, N. Y. Saction: CA08:002 Publ Class: TECH REP Users In S. MIIS, Ps. Rep. Coden: XPBCA Publ: 77 Issue: Ps-274635, Pages: 87 Pp. Citation: Gov. Rep. Announce. Index (U. S.) 1978, 78(9), 181 Avet: MIIS

Seturalisation of polymeles areastic hydrocarbons contained on the object of polymeles areastic hydrocarbons contained of the contained of the

 A mea generation of monitors for polymotear aromatic A mea generation of monitors for polymotear aromatic flydrogarbons from synthetic fuel production.

Author: Gammage, R. E.; Vo-Dinh, Tuan; Hauthorne, A. R.; Thorogate, J. H.; Parkinson, W. W.

Location: Health Saf, Res. Div., Oak Ridge Natl. Lab., Oak Ridge, ferm.

Section: CAGSOG2, CAGSIKK, CAGTSIKK Publ Class: JONONAL Section: CAGSOG2, CAGSIKK, CAGTSIKK Publ Class: JONONAL Section: Garcinog, - Compr. Surv., Coden: CCSLDL Publ: 78 Series: 3 Issue: Polymicl. Aromat. Hydrocarbons Pages: 185-4.

Identifiers: polymiclear arom hydrocarbon air monitoring, fuel processing polymiclear hydrocarbon monitoring.

Cardingenesis - A Comprehensive Survey, Vol. 3:
Cardingenesis - A Comprehensive Survey, Vol. 3:
Cardingenesis - A Comprehensive Survey, Vol. 3:
Polymidaer Analysis, Chemistry, and Biology
Author: Johns, Peter W.: Freudenthal, Raiph I.: Editors
Location: USA
Section: CA004007 Publ Class: 800K
Coden: BOOKA7 Publ: 78 Pages: 487 pp.
Coden: BOOKA7 Publ: 78 Address: 487 pp.
Identifiers: Book polymicles aros Pydrocarbon carcingen

Becords consocious chromatographic analysis for Detectors for liquid chromatographic analysis for polymusiaer areasis (Mydrocarbons Author: Christensen, B. G.; May, W. E. Location: Anal. Chem. Div., Matil. Bur. Stand., Washington, D. C. Section: CA08002 Publ Class. JOURNAL Journal J. Liq. Chromatogr. Coden: J. Chomatogr. Coden: J. Chromatogr. Coden: J. Chromatogr. Coden: J. Chromatogr. Coden: J. Chromatogr. J. Chromatogr.

Senigrantitative and quantitative methods for the debendentiative and quantitative methods for the debendents of pelymelear artmatic hydrocarbons in bitamination and perturblame.

Location: Bahina, M. M.
Location: USSS
Section: Location: Coopers Publicias: Journal Coofic.

Journal: Tr. Sib. Mauchno-lasted. Inst. Gool. Goofic.

Miner: Syr'ya Coden: 151644, Publ: 76 Series: 231, Papes: 144-8 Language: Buse
Identifiers: patroleum arem luminescence detn. bituminoid arem luminescence detn.

Bedissi CAGEGOZOIISSIS

Betweeten for a queene chlorination reaction products of polymedian artestic hydrocarbane by reversed phase high performance liquid chromategraphy gas chromategraphy. Resemble flying the characteristic hydrocarbane by reversed phase high performance liquid chromategraphy gas chromategraphy. R. J.; Lukkonen, E. d. Telean, E. H.; Exportann, H. L.; Caple, R. Location: Dep. cham., Univ. Histoperann, H. L.; Caple, R. Location: Dep. cham., Univ. Histoperann, H. L.; Caple, R. Location: Caple, R. Series: Andrews and Caple of Caple

Melector consistency.

The use of a combination of altraviolat and fluorescence defectors for the selective detection and quantitation of polymeister around the selective detection and quantitation of polymeister around the following polymeister around the following polymeister around the following polymeister around the following processing for the following foll

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Selected of polymentar aromatic hydrocarbons by reversed phase thin-layer chromatography
Author: Shiralahi, Voshiko; Vamashita, Tamko; Shirotori,

Location: Mati. Inst. Public Health, Tokyo, uspen Section: CAGGODOM Publ Cleas: JOURNAL Journal: Else! Kagan Journal: Else! Kagan Section: 23 Issue: 8 Pages: 210-13 Language: Japan Identifiers: polymiclear arom hydrocarbon chromatog. 1

layer chromatog arom hydrocarbon, reversed phase thin layer chromatog

dentification of environmental polymusicar arosatic Mydrocarbone by pulsa fourier-transform preten nuclear magnetic resenance spectrescopy.

Author: Barile, K. D.; Lee, M. L.; Movotny, M. Location: Deep Phys. Chem., Univ. Leads, Leads, Engl. Section: CA004001 Publ. Class: Julibrat.

Location: Analyst (London) Coden: AMALAD Publ: 77 Series: 102 lease: 1219 Pages: 731-8

Identifiers: polymuclear arom hydrocarbon identification.

New Fourier transform hydrocarbon, all polymuclear arom hydrocarbon, amerithman smoke polymuclear hydrocarbon, amerithman

Polymachaer aromatic hydrocarbons in coal - identification by their x-ray excited optical luminascence.

Author: Woo, Ching S.; D'Silva, Arthur P.; Fassel, Velmer A.; Gestreich, Geogory J.

Location: Ames Lab., lowe State Univ., Ames. lowa.

Section: CAGGSMIS, CAGGSMIX, Publ Class: JUNHAL Journal: Environ. Sci. Technol. Coden: ESIHAG Publ: 78

Series: 12 Issue: 2 Pages: 173-4

Identifiers: arom Mydrocarbon coal x ray, luminascence arom Mydrocarbon coal, safety carcinogen coal datn.

0806578; CAO081006578;; Ophn-pore for collection and precencentration of polymeter aromatic hydrocarbons from water. Author: Mavratil, James D.; Slavers, Robert E.; Walton, Harold F.

Ser les: Grethera Location: Booky Flats Plant, Bockwell Int., Golden, Colo-Section: CA051002, CA037XX Publ Class: Journal Journal Andi-Chas. Octor: ANDIAN Publ: 77 Serie deural: Andi-Chas. Octor: 2260-3 Identifiers: polywrethene pyrene resoval water, urathe polymer parene resoval water, urathe

87) User 3631 2spr81 DIALOG File104: CA Search - 77-79/Vol 91(26) (Capr. Am. Chem. Soc.) (Item 64 of

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A SACE.

Determination of the agreems solubility of polymolear sensition by the agreems solubility of polymolear sensition by a coupled column liquid chromatographic technique Author: May, Willie E.; Wastk, Stanley P.; Freeman, David H. Location; Inst. Mater. Res., Nati. Bur. Stand., Washington,

Section: CAGEBOOI, CAGEBUXX, CAGEIXXX Publ Class: JOURNAL JOurnal: Anal. Chem. Coden: ANCHAN Publ: 78 Series: 50 Issue: 1 Pages: 176-9 Identifiers: soly detn area 11q chromatog, polymuclear area soly detn chromatog

Polymedian argentic content of heavy duty diesel engine makes gases
Author: Spind, R. S.
Location: Gulf Res. and Dav. Co., Pittsburgh, Pa.
Soction: CAGGOOI, CAGGOOIX
JOURNAL! U. S. MIS, PE Rep.
Coden: XPERCA Publ: 77
Issue: PE-267774, Pages: 50 pp.
Citation: Gov. Rep. Announce. Index (U. S.) 1977, 77(18).

Identifiers: polymuclear area detn exhaust gas, hydrocarbon erom detn exhaust gas Avail: MTIS

Advances in the enelysis of polymoster arematics Advances in the enelysis of polymoster arematics Author: Sewerland, H. D.; Stadelhofer, J.; Thoms, R.;

Ander, H.
Location: Assignments A.-G., Castrop, Ger,
Location: CAGBODG Publ Class: Unimited.
Journal: Campend. Bitch, Ges. Mineralcolviss. Kohleches.
Coden: Caging Publ: 78 Series: 78-77 Issue: 2

Coden: Codible Publ: 76 Series: 78-77 Issue: A Depart Sel-7 Language: 6a-7 Issue: A Identification anal, gas Identification polymeiser area hydrocarbon anal, gas chromatog polymeiser area hydrocarbon, 14 chromatog hydrocarbon, 14 chromatog hydrocarbon.

Obsolves CAO8724183286A
Chamically-bonded mainteellane stationary phase for the high-perference liquid obrematographic saperation of polymenter areasts companies
Author: Wise, S. A.; Chester, S. N.; Hertz, H. S.; Hilpert, R. R.; May, W. E.
Location: Inst. Meter. Res., Matt. Bur. Stand., Mashington, B. C.

Publ Class: JOHNHAL

Section: CADBODD4

.umittiers: aminosilane bonded phase 11q chromatog. etationery phase aminosilane 11q chromatog. high performance 11q chromatog. polynuclear area 11q chromatog. hydrocarbon aligh 11q chromatog. Series: Publ: 77 Journal: Anal. Cham. Coden: ANCHAM 49 Issue: 14 Pages: 2306-10 Identifiers: asinosilane bonded (

Make in the analysis of polymusian aromatics
Author: Sauariand, H. D.; Stadelhofer, d.; Thoms, R.;
Zander, M.
Location: Rustgerswerke A.-G., Castrop-Rauxel, Ger.
Section: GAOSIODI, CAOBOXXX Pubi Class: UDURAAL
JOURNAL: Erdost Kohle, Erdost, Pubi Class: UDURAAL
JOURNAL: T. 7 Saries: 30 Issue: 6 Pages: 215-16
Language: Gar
Language: Gar
Language: Gar
Journal: Erdost Mydrocarbon analysis, Chromatog
polycyclic arom hydrocarbon celumn chromatog arom hydrocarbon
gas chromatog arom hydrocarbon
fluorescence spectroscopy
fluorenthens deriv, NWM carbon 13 arom hydrocarbon

Adsorbent for polyminates aromatic compounds
Author: Stelling, David L.; Muckins, Jomes N.; Saith, 87154137 CAO8720154137H

Location: USA
Soction: CA046001, CA360XXX Publ Cleas: PAT
Journal: U. S. Pat Appl. Coden: KAXXAV Publ: 761018
Pages: 7 pp. Avall. NIIS.
Patent No: 733500 Applic No: 733500 Date: 761018
Assignme: United States Dept. of the Interior
Identifiers: charcoal polyurathane adsorbent, polynuclear
aroa compd adsorbent, chlorobenzodioxin dein, dioxin deriv

#7089625 CAO#7120896282
The high pressure liquid chromategraphy and its application to the seperation of polymosteer areastic hydrocarbons in atmospheric data and burning residuas
Author: Lopez, M. C.
Location: CEM, Commis. Energ. At., Granobis. Fr.
Section: CAO#0001, CAO#00XXX Pub. Class: TECH RF.
Murnal: Report Coden: D2MEPU Publ: 78 Issue: Cital-14078. Pages: 44 pp. Language: Fr.
Cital-1601: MIS Atmindex 1976, 7(7), Abetr. Mo. 232667
Avel: 1815

residue Identifiers: sepn polycyclic hydrocarbon chrosetog, polycyclic hydrocarbon chrosetog, combustion ri hydrocarbon chrosetog

Publ: 77 Section: CA00400; Publ Class: JOURNAL Journal: Environ. Sci. Technol. Coden: ESTHAG Series: 11 Issue: 7 Pages: 682-6 Identifiers: benzopyrene removal water polyurethans

Platescence characterization and identification of Platescence characterization and identification of polymerlear areasts byteschane in shale all Author: thrithies, E. J.; Schabron, J. F.; Faaster, J. D.; Institildean, D. H.; Poulson, R. E. Location: Dep. Chem., Univ. bytesing, Laranie, Byo. Section: CADS 1014, CAGBOALX Publ Cleas: JOURNAL JOHNAL Series: 89 | Saue: 2 Pages: 377-82 | Identifier: fluoracence apectrometry arom hydrocarbon, chrostog arom hydrocarbon, chrostog and data data data shale oil, pyrene data shale oil, benzopyrene data shale oil

87027950 CAO87040279501
Determination of polymolese aromatic hydrocarbons by thin-layer chromatography
Author: Wikeleeve, M. M.; Koroleva, K. I.; Korunov, A. A.

Location; USSR Section; USSR Section; USSR CAMBIXXX, CAMBOXXX Publ Class; JOURNAL JOURNAL: S. Vess. Mauchno-Issled. Produting-Thist. Elektroupo! Tryin Isda ! Coden: TMIZOC Fubl: 75 Secies: 3, Pages: 125-30 Language: Russ Identifiers: phananthrens emission binder, fluoranthens

emission binder, polycyclic hydrocarbon emission binder, bentzogyrana emission binder, sethylcholanthrene emission binder, cholanthrene enthyl emission binder, ter binder polycyclic hydrocarbon, pitch binder polycyclic hydrocarbon, pitch binder polycyclic hydrocarbon, anthracana oil binder emission, carbon graphite article emission, chromatog polycyclic arom hydrocarbon, thin layer chromatog hydrocarbon

Defermination of polymodear aromatics in yeast produced by paraffin fermantation and n-phydrocarbon feedstocks Author: William William and n-phydrocarbon feedstocks Location: Welliams, E. L.; Morris, M. S. Location: Gulf Res. and Dev. Co., Pittshungh, Pa. Section: CAO16001 Publ. Class: Unknhall and Chem. Soc. Coden: ACPCAT Publ: 75 Series: 20 Issue: 4 Pages: 829-37 Identifiers; polymodear arom single cell protein

aromatic .. : 86:84:15 CAOS628:941:90
Tentative sethod of analysis for polynuclear hydrogarbons in automobile schaust
Author: Savicki, E. Location: USA,

Section: CAOSOO1, CAOSOXX Publ Class: JOURNAL Journal: Health Leb. Sci. Coden: HLSCAE Series: 11 Issue: 3 Pages: 228-39 Identifiers: arom hydrocarbon dein exhaust

Section: CA069001, CA081XXX, CA080XXX Publ Class: JOURNAL Journal: Health Lab. Sci. Coden: HLSCAE Publ: 74 Sectes: 11 Issue: 3 Pages: 218-27 Identifiars: arom Pydrocarbon detn coke oven SelBeits CAOSSESSISTISP Tentative mathod of analysis for polytuclear Author: Savick: E. Location: USA

87) User 3631 2apr81 78 of DIALDG File104: CA Search - 77-79/Vol 91(26) - (Copy. Am. Chem. Soc.) (Item

CA08622 164856K

Philhalisidepropylishers - a new changeally bonded stationary phase for the determination of polymorlear aromatic hydrocarbons the high-pressure liquid chromatography author: hunt, D. C.; Mild, P. J.; Crosby, N. T. Location; Lab. Gov. Chem., London, Engl. N. T. Location; Lab. Gov. Chem., London, Engl. N. T. Section: CAGEGOOG, CAGGGRAY, Publ Class: JOURNAL Journal: J. Chromatogr. Coden: JOCARA Publ: 77 Series: 130. Pages: 320-3 Identifiers: high pressure liq chromatog, propylsilane stationary phase liq chromatog, propylsilane philas sidophase liq chromatog, silane phitalisidopropyl phase liq chromatog, silane phitalisidopropyl phase liq chromatog, solventlane chromatog, polymorlear arom hydrocarbon liq chromatog, massel analysis polymorlear arom hydrocarbon, benzofluoranihane detection massel.

100217 CAGES2160217E
Investigation on a long-term collection of polymician arematic hydrocarbone in environmental air
Author: Matsuchita, Hidetauru; Arashidani, Kelichi; Handa,

Location: Mati. Inst. Ind. Health, Kawasaki, Japan Section: CAGGGOI, CAGGGXX Publ Class: JOSENIAL Journal: Bunsaki Kagaku Coden: BNSKAK Publ: 76 Series: 25 Isawe: 6 Pages: 415-17 Language: Japan Identifiers: golycyclic area hydrocarbon sampling air, glass filter area hydrocarbon retention

96160216 CAD6622160216D Vacuum sublimetion method for extraction of polynuclear aromatic hydrocarbone from elrhorne particulates
Author: Mateuahita, Hidelauru; Arashidani, Kelichi; Hayashi,

Location: Metl. Inst. Ind. Health, Kawsseki, Japen Scrien: CAOSSON: CAOSONX Publ Cless: JOURNAL Journal: Bursaki Kaphu. Coden: BNSAN Publ: 78 Series: 25 Issue: 6 Papes: 412-15 Language: Japen Identifiers: area hydrocarbon vacuus subilastion app, polycyclic area hydrocarbon extn perticulate

Author: Mateuahita, Hidatauru; Arathidani, Keilchi; Koyano, Michiko; Handa, Takarhi Location: Mail. Fre: Ind. Health, Kausaki, Japon Section: CAGEGCT; «ADGOXX Publ Cleas: JOURNAL JOHNAL JOHNAL JOHNAL TO BE 1 I laue: I Pages: 44-53 Language: Japon Identifiers: gesoline benzopyrane date, arene date petroleum 66197966 CAC6622187968V A Simple rapid analysis of polymenicar promotic hydrocarbons in gaselina

oil. arom polycyclic hydrocarbon datn

36

Analysis of polymadian aromatic hydrocarbons in the respirately exticoment.

Author: Strummann, Klaus D.; Hoffmann, Dietrich Location: Maylor Dana Inst. Dis. Prev., Am. Health Found., Valhalla, N. Y.

Section: CA050002. CA011XXX Publ Class: JOURNAL Journal: Carcinog. - Compr. Surv. Coden: CCSUDL Publ: 76 Saries: 1, Pages: 283-97 Identifiers: tobacco smoke carcinogen, polycyclic arom hydrocarbon smoke, chrysene cigaret smoke, fluorathere cigaret smoke

An integrated approach to the analysis of air-pollutant polymolear arematic Mydroanabons
Author: Bartie, M. D.: Lee, M. L.: Novotny, M. Location: Sch. Chem., Univ. Leeds, Leeds, Engl.
Location: Sch. Chem., Univ. Leeds, Leeds, Engl.
Sociton: CADBOOGS, CAODAXXX, CAOSSXXX Publ Class: JOURNAL JOURNAL Sociton: CADBOOGS, CAODAXXX, CAOSSXXX Publ Class: JOHN Papps: John P polymuciesr aros

Author: Katz. Morris: Pierce, Ronald C. Location: Cent. Res. Environ. Qual., York Univ., Toronto, Section: CA059002 Publ Cless: JOURNAL Journal: Carcinog. - Compr. Surv. Coden: CCSUDL Publ: 6 Series: 1, Pages: 413-28 Identifiers: polycyclic aroa hydrocarbon distribution, perticle size polycyclic hydrocarbon of polymoless as particle size of 8608524 CAO861408524F Quantitative distribution hydrocarbons in relation to particulates

Becommendation of polymuster areastis hydrocarbons (PAH)
Author: Stepanova. M. I.
Location: Derishink. Filisi, Gob. Neuchno-Issled. Inst.
Location: Constitute Gazov, Dzerzhinsk, USSR
Section: CAGSOOI. CAGGOXIX. Pabl Cless: DUBNAL
Journal: Pros. Sant. Ochistka Gazov Coden: PSGADK
Publ: 76 Issue: 2 Pages: 30-1 Language: Russ
Identifiers: polymuster area hydrocarbon waste gas

Medesone Cacestocestoest
Microanalytical methods for polymiciaer aromatic
Mydrocarbons in environmental air
Author: Matsuerita, Hidetauru
Location: Matl. Enst. Brd. Health, Kaussell, Japan
Sertion: Codecolo, Cacoboxxx Publ Class: Jolieka,
Journal: Taiki Geen Kenkyu Codon: Toktav Publ: 76
Series: 10 Ispue: 8 Pages: 723-31 Language: Japan
Identifiers: review polymiciaer arom hydrocarbon air

Belorates coosciolosessy and to the constraint of caralless and belorates and considered sand considered sand considered sand considered sand care pelymenter aromatic hydrocarbons Author: Nation H. 1. Grisst, W. H.; Guerin, N. R. Contion: Anal. Chem. Div., Oak Ridge Hatl. Lab., Oak Ridge, Section: CAGO-001 Publ Class: Journal. Trace Subst. Environ. Health Coden: Publing Lab.: 7 Scies: 9, Pages: 281-9 Identifiers: 100-000 sanks arom hydrocarbon detn, coal product arom hydrocarbon detn.

1 of 45) User 3631 2apr81 Print 13/2/1-45 518100 file3: CA Search - 1872 thru 1976 (Copr. Am. Chem. Soc.) (Item

Analy:idal methods for polynuclear prometic hydrocarbons in crude ells, healing ells, and marins tissues Author: Pencifor, R. J.; Brown, R. A. Location: Anal. Inf. Div., Exxon Res. and Eng. Co., Linden, Coden: Section: CAGS1001, CAGS0000 Publ Class: C Journal: Proc. - Conf. Prev. Control 011 Pollut. MPAX Publ: 75 Pages: 103-13 Mahington, D. Publisher: As Pet. Inst. Address: Mahington, D. Identifiers: polynuclear area hydrocarbon petroleum CA08526 1949737

Howard هر چ 3 Athens Scolation, identification, and quantitation polymaclear areasts hydrocarbons in tobacco smoke Author: Severson, Ray F.; Snok, Haurice E.; Higman, C.; Chortyk, Greates T.; Akin, Frank J. Location: Job. Health Res. Lab., Agric. Res. Cent., Section: CA011007 Publ Class: J Journal: Carcinog. - Compres. Surv. Coden: CCSUDL 1 Series: I, Pages: 283-70 Identifiers: tobacco seoke aromatic hydrocarbon CA08525189419N 62 1894 19

Defermination of polymorlear aromatic hydrocarbons by anodic differential pulse veltametry at the glassy carbon electrode in sulfelane and ecatomitrile as solvents conton: Dep. Ches., Univ. Pittsburgh, Pittsbur CA08524 IB 1842J

Authors of polymorae aromatic hydrocarbons in automobile exhaust set polymorae aromatic hydrocarbons in automobile exhaust by supercritical field offromstography
Author: dentoft, R. E.; Gouw, T. H.
Location: Chevron Res. Co., Richmord, Call f.
Section: CAOSOOO; CAOSOOO, CAOSOOO Publ Cleas: Judurnal: Anal. Ches. Coden: AMCHAM Publ: 76 Series: A Begas: 21859
Identifiers: anhaust arom polyminlar hydrocarbon detn, chromatog supercrit polymuclear hydrocarbon detn,

85172333 CAO8523172333G

8

Market Severation of polymiclear aromatic hydrocarbons in tobacco smoke Author: Saverson, R. F.; Snook, M. E.; Arrandale, R. F.; Chortyk, O. F. Location: Tob. Lab., ARS, Athans, Ga. Location: CA004001, CA011000, CA080000 Publ Class: J. Journal: Anal. Cham. Goden: ANCHAM Publ: 76 Series: J. Jasses: 13 Pages: 1866-72 Identifiers: tobacco smoke hydrocarbon gas civomatog

Detaction and determination of polynuclear aromatic Mydrocarbons by luminascence spectrometry utilizing the Mydrocarbons by luminascence axotistion spectra at 77 degree.K. Part III. Luminascence axotistion spectra CA08522171302J

Author: Farooq, R.; Kirkbright, G. F. Location: Dep. Chem., Imp. Coll. Sci. Technol., London,

Engl.
Section: CA080005, CA073200 Publ Class: J
Journal: Analyst (London) Coden: ANALAD Publ: 76
Series: 101 Issue: 1204 Pages: 866-73
Identifiers: polymaclear arom hydrocarbon detection,
luminescence excitation polymaclear hydrocarbon, spectrometer
luminescence excitation Sipolekii

85165904 CA08522165904R Analysis of polynuclear aromatic hydrocarbons in complex mixtures

Author: Lee, Milton Lefayette
Location: Indiana Univ. Bloomington, Indiana
Saction: CA055009, CA025000
Saction: CA055009, CA02500
Coden: DA888A Publ: 75 Pages: 251 pp.
Citation: Diss. Abstr. Int. 8 1976, 36(11), 5548
Asil: Kerox Univ. Microfillas, Ann Arbor, Mich., Order No.

Identifiers: arom hydrocarbon datn air, carcinogen detn air 76-11,369

A chromatographic analysis for polynuclear aromatic hydrocarbons in small quantities of eigeret smoke condensate Author: Severson, R. F.; Snook, M. E.; Chortyk, O. T.; Arrendale, R. F.

Publ: 76 Location: Tob. Lab., ARS, Athens, Ga. Section: CA011007, CA009000 Publ Class: Juournal: Beiter: Tabakforsch. Coden: RETAAV Pries: 8 lasue: 8 Pages: 273-22 Identifiers: tobacco smoke hydrocarbon analysis

CA085 14098579Y 85098879

Gass chromatography ass spectromatric and mudiar magnatic resonance determination of polymudiaar aromatic hydrocarbons in airborne particulates.

Author: Lee, M. L.; Hovotry, M.; Bartle, K. D. tocation: Dap. Chem., Indiana Univ., Blocomington, Indiana Section: CAGSODI, CAGBOODO Pubi Class: J. Journal: Anal. Chem., Coden: ANCHAM Pubi: 76 Series: Journal: Anal. Chem. Coden: ANCHAM Pubi: 76 Series: Hassus: 11 Pages: 1866-72 Identifiers: hydrocarbon data alrborne particulate, arom hydrocarbon particulate, mass spectrometry arom hydrocarbon particulate.

High-pressure liquid chromatography of polynuclear aromatic hydrocarbon constituents of smoke Author: Heberter, A. F.; Chortyk, O. T. Location: Tobacco Lab., ARS, Athens, Ga. Section: CADIIOO7 Publ Class: U. Coden: RAISDZ Publ: 78 Journal: Rocant Adv. 100, Sci. Coden: RAISDZ Publ: 78 Series: I Issue: New Yech. Smoke Chem. Phys. Pages:

Prom Smoke. hydrocarbon tobacco smoke raview

Becases Cacesizosesos
Detection and determination of polynuclear aromatic
Detection and determination of polynuclear aromatic
Shydrocarbons by luminascence specificanty utilizing the
Shydriviti effect at 77 degree.K. Pert II. An evaluation of
excitation sources, sample cells and detection systems
Author: Causey, B. S.; Kirhbright, G. F.; De Lima, C. G.
Location: Dep. Chem., Imp. Coll. Sci. Technol., London,

Section: CAGEGOOG Publ Class: J Journal: Analyst (London) Coden: ANALAD Publ: 78 Jeries: 101 Isaus: 1202 Papes: 367-78 Identifiers: polynuclear hydrocarbon data luminescence, arom hydrocarbon data luminescence, Luminescence appetitionality hydrocarbon instrumentation, excitation source luminescence hydrocarbon, cryostat cell luminescence hydrocarbon

Selective monitoring of polynuclear areastic hydrocarbons by high pressure liquid chromatography with a variable wavelength detector: Krstulovic, Ante M.; Rosie, Douglas M.; Brown, Krstulovic, Ante M.; Rosie, Douglas M.; Brown, CA085 10067425R

Location: Dep. Chem., Univ. Rhode Island, Kingston, R. J. Section: CA089001, CA047000, CA080000 Publ Class: J

Phyllis R.

Ser tes: polynuclear arom arom hydrocarbon, Publ: 76 Journal: Anal. Ches. Coden: ANCHAM 18 Issue: 9 Pages: 1383-6 Inching Identifiers: chrosatog soultoring hydrocarbon, UV detector chrosatog sovitoment polynuclear area analysis

Publ: 76 tocation: Imp. Coll., London, Engl. Section: Code1002 Publ Class: J Journal: Water Res. Coden: WATRAG Issue: 3 Pages: 207-12 9

Ser tes:

Identifiers: arom hydrocarbon detn water

CA0850704 1646P 85041646

Gas chromatographic analysis of polymuclear aromatic hydrocarbons in shellfish on short, wall-coated glass capillary columns

Author: Onuska, Francis I; Wolkoff, Aaron W.; Comba, Michael E.; Larese, Richard II.; Novotny, Wilos: Lee, Milton L. Location: Canada Cont. Interv. daters, Burlington, Ont. Section: CAOGAGOI, GASGOGO Publ Class: J. Journal: Anal. Leit. Cocky: AMALBP Publ: 76 Series: 9 Issue: 6 Pages: 451-60

identifiers: arom hydrocarbon gas chromatog. shellfish arom hydrocarbon detn

84189993 CAO8426.88083F X-ray excited optical tuminescence of polynuclear aromatic hydrocarbons

Series: Author: D'Silve, A. P.; Destreich, G. J.; Fassel, V. A. Location: Ames Lab., Town State Univ., Ames, John Section: CAGGOOG, CAGGOOG Publ Class: J. Journal: Ansl. Chem. Coden: ANCHAM Publ: 76 Section: Ansl.

identifiers: x ray excited optical luminescence, luminescence detection polynucleer arom hydrocarbon, perylene luminescence detection, benzopyrene luminescence detection, coronene luminescence detection. Pages: 915-17 ldentifiers: =

84071660 CAO8411071660K Reselution of polynuclear aromatic hydrocarbons by packed column 82.C Section: CADIBOT, CADORDO Publ Class: July Cadoria MALBP Publ: 78 Series: Esue: 12 Pages: 949-57 Identifiers: Panzanthracana tobacco seoke, phenanthrana dath tobacco seoke, chromatog hydrocarbon tobacco seoke Author: Griest, W. H.; Kubota, H.; Guarin, M. R. Location: Anal. Chem. Div., Oak Ridge Nati. Lab., Oak Ridge, -Section: CA055002, CA050000 Publ Class: J
Journal: Ann. Occup. Hyg. Coden: A0HYA3 Publ: 78
Series: 18 Issue: 3 Pepes: 196-206
Identifiers: dust polynuclear aron hydrocarbon, benzopyrana
dust circaetog, benzoprylana dust chrosatog, anthracena dust
chrosatog, benzochrysena dust chrosatog

Adiabata conditional in the environment. It betwingles aromatic hydrocarbons in the environment. It betwinstantente pelynuclear aromatic hydrocarbons in water by mass fregmentegraphy.

Author: Matsuchima, Hajima; Harya, Takahima
Location: CADB, Chem.; Tokyo Matrop. Univ., Tokyo, Japan Section: CADB1022 Publ Class: Juliv., Tokyo, Japan Section: CADB1022 Publ Class: Juliv., Tokyo, Japan Series: 24 Issue: B Papes: 506-11 Language: Japan Identifiers: carcinogen hydrocarbon dein fiver water. Fluoranihram dein river uster. benzpyreim dein river uster.

S4045362 CAD84080493621

Thin-layer chromategraphic determination of polymolear aromatic hydrocarbone (in waste gases)
Author: Wholeave, N. N.; Koroleva, K. I.; Korunov, A. A.

Selected CAOSAISIDGESE Gas chromelography/mass spectrometric and rustear magnetic resonance spectrometric studies of carcinogenic polynuclear aromatic hydrocarbons in tebaco and marifusa smoke condensates Author: Lee, M. L.; Movotny, Milos; Bartle, K. D. Location: Dep. Cho. Indiana Univ., Bloomington, Indiana Section: CAOSAGO Publ Class: J

Author: Lee, M. L.; Movotny, Milos; Bartle, K. D.
Location: Dep. Chee., Indiana Univ., Blocaligton, Indiana
Section: CA004007 Publ Chas: J
Journal: Anal. Chem. Coden: AMCHAM Publ: 76 Series:
48 Issue: 2 Pages: 405-16
Identifiers: tobacco smoke arom hydrocarbon, marijuana smoke
arom hydrocarbon

B3188012 CA08322188012W Superorities fluid chromatography of poly

chroma tog

Location: USSR
Section: CA08001, CA08000 Publ Class: Juburnal: Tr. Vesa. M.-1. I Proekt.-Tethnol.
Clastrougoi'n. Exdeli Coden: 044005 Publ: 75
2 Pegas: 125-30 Language: Russ
Citation: Ref. Zh., Khim. 1976, Astr. No. 18990
hydrocarbon waste gas. Chr.

Signaturalities fluid chromatography of polymuclear aromatic hydrocarbon.
Author: Futita, Kazunori; Shimokoba, Ikuo; Makazima, Frmito Location: Mitachi Res. Lab., Mitachi, Ltd., Hitachi, Japan Section: Ghobood, CAOSiODO Publ Class; U dournal: Mippon Kagaku Katahi Codes: Makes Publ: 75 Issue: 8 Pages: 1348-81 Language: Japan Identifiers: supercrit fluid chromatog arom hydrocarbon, chromatog polymuclear arom hydrocarbon, chromatog polymuclear arom hydrocarbon, chromatog chromatog, phenanthene chromatog, pyrenchromatog, chrysene chromatog, triphenylene chromatog, coaltar supercrit fluid chromatog

Accessor 2 CACE41208502R High-pressure liquid chromatography of polynuclear aromatic Muthor: Heaberer, A. F.; Snock, M. E.; Chortyk, D. T. Location: Tob. Leb. A83, Althana, M. Location: GOOGODI Publ Glass: J. Journal: Armal. Chim. Acta Coden: ACACAM Publ: 78 Section: Besser: Propres: 303-9 Identifiers: arom hydrocarbon liq chromatog, cigaret seoke condensate 1iq chromatog Maintage capazingation between the determination of trace assumts of pelymeiser areastic fortherestrong Author: Mirkelight, 6. F.; Be Lina, C. G. Location: Cham. Bep., Rep. Coll., London, Engl. Section: CADOMOSI Publ Class. J. Coden: PAYCAL Publ: 74 Series: 19 Toc. Soc. Anel. Cham. Beps: 86-60 Identifiers: area hydrocarbon deth Shoolskii effect

Biodician Chomical of the fate of polymolear aromatic bydrocarbons in natural vater systems for the cast in natural vater systems for the cast inclinate, Paul B.; Smootink, Vernon L. Gention: Dep. Civ. Eng., Univ. 111 inclinate, Urbana, 111. Section: Cadebook, Cadebook, Publ. Class. J. dep. Civ. Eng., Univ. 111. Urbana-Chapsign, Water Resour., Coni. Coden: IUNIAM Publ: 74 Series: 80, in natural water, benzpyrene decompn arom hydrocarbon decompn decompn natural water, br Resour. Cent. Pages: 56 pp. Identifiers: Denzanthracamo Natural water

Trade pelymuclear aromatic hydracarbon analysis
Author: Hearni, E. B.; Flacibach, H.
Location: Bur. Foode, Code Grug Ade., Washington, B. C.
Section: CA017000, CA004000 Publicless; C.
Journel: Contrib. Chem. Food Supplies, Invited Sel. Contrib.
Pp. Symp. Coden: 28XVAZ Publ: 74 Pages: 209-25 Pap. Symp. Goden: 28XVAZ Publ: 74 Pages: 209-28 Meeting Date: 73 Publisher: Butlerworth Address: London, Engl Avall: Morton, I.; Mades, D. M Identifiers: review hydrocarbon dein food, carcinogen dein food review 82 1838 15

Preliminary results on the use of Tenax for the extraction of positioning results on the use of Tenax for the extraction of positioning the position of the present of the state of the sta

Determination of four- and five-ring condensed hydrocarbon.

II. Analysis of polymeisar aromatic compounds in n-paraffin feed oil for yeast formantation.

Author: Wolfine, Edger L.

Location: Golfool, CA028000, CA023000 Pubi Class: J
Section: CA018001, CA028000, CA023000 Pubi Class: J
Section: CA018001, CA028000, CA023000 Pubi Class: J
Source: J Species: 20 Coden: JAFGAU Pubi: 75
Sectes: 21 Issue: 2 Peges: 226-9
Identifiers: arom analysis paraffin yeast protein

Before the contribution of fear- and five-ring condensed hydrocarbons.

1. Analysis of palvindlast areasite hydrocarbons in yeast predated by grouth see both in-hydrocarbon and dixtross feeds Author: Redinits, Edgar L.; Morris, Matthew S. Location: Gulf Res. and Dev. Co., Pittsburgh, Pa. Section: CA018001, CA038000 Publ Class: J. Journal: J. Agric. Food Chem. Coden: JAFCAU Publ: 78 Series: 23 Issue: 2 Pages: 22:6
Identifiers: arom hydrocarbon anal yeast CA08221137656F 82137656

Profiles of the pelymedian aromatic fraction from engine and nitrogeness by Explination grounding and nitrogeness and nitrogen

Migh resolution &C. (gas-liquid chrometography) profiles of urban air pelfutant pelgrandian for the pelgrandian for the pelgrandian for the following the following section: Bartia, K. D.; Lee, M. L.; Movofry, M. Location: Dep. Chem., Indiana Univ., Sloomington, Indiana Section: CAOSOO2. CAOSOOO Publ. Class: J. Johnson Publ. Class: J. Cubertal: Int. J. Environ. Anal. Class: J. Coden: LuEAA3 Publ: 74 Series: 3 Issue: 4 Pages: 348-56 Identifiers: aron hydrocarbon detn air

\$2137657 CA082211378878

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APPENDIX B
PROJECT QUALITY CONTROL PLAN

PROJECT QUALITY CONTROL PLAN

APPENDIX B

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1.0 INTRODUCTION

This document describes the quality control procedures to be used for the methods development and analysis efforts required in this project. The plan described in the following six sections complies with the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) Quality Assurance (QA) Program and with Environmental Science and Engineering, Inc. (ESE) policy. ESE supports an active, comprehensive QA program within its Technical Operations Division, which is described in the Division's Operations Manual. A major focus of the ESE QA program is the development of a project Quality Assurance/Quality Control (QA/QC) plan describing the specific application of ESE procedures to control and monitor any project.

The specific objectives of this plan are to describe in detail the process for controlling the validity of the data generated for documentation of the analytical methods developed. The Project QA Plan provides a mechanism for documentation of the limits of precision, accuracy, and sensitivity of all analytical systems generating data.

The prospective analytical approach for each analyte will be described in the proposed method reports which will be submitted for approval as required by USATHAMA. The analytical systems controls and data validation procedures described in this Quality Control Plan will be employed to ensure valid, properly formatted data defining the precision, accuracy, and sensitivity of each method. Reports will also be submitted to USATHAMA documenting the methods in both natural and standard media.

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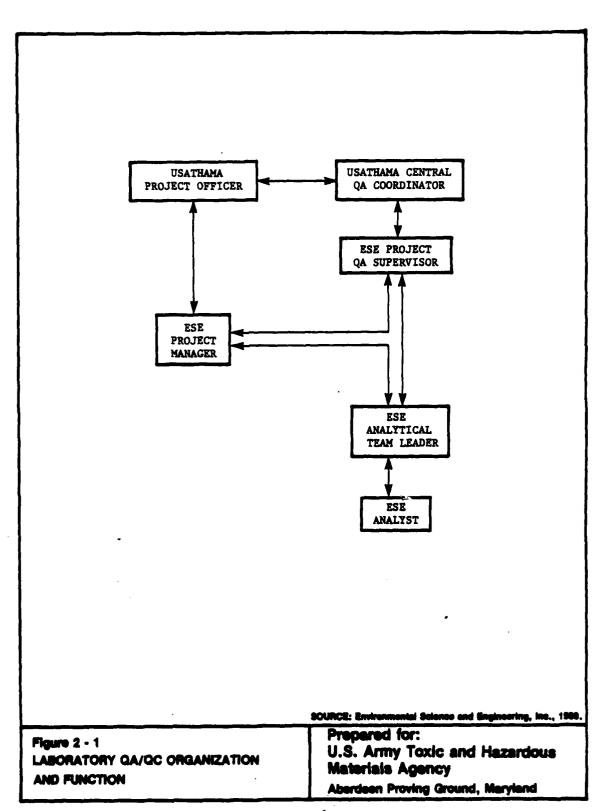
2.0 ORGANIZATION AND RESPONSIBILITIES FOR QUALITY ASSURANCE

The Quality Control Plan functions according to the USATHAMA central-laboratory/field-laboratory concept. ESE acts as the field laboratory which is monitored by the USATHAMA Central Laboratory QA Coordinator. The overall QA/QC organization to provide valid analytical methods to the commander of USATHAMA is shown in Figure 2-1. The function of the plan and QA responsibilities of each of the project participants are outlined in the following subsections.

2.1 OVERALL PLAN FUNCTION

Figure 2-1 depicts the manner in which the ESE Project Quality
Assurance/Quality Control (QA/QC) Supervisor monitors the conduct of the project. In this position, the QA/QC Supervisor is not directly subordinate to snyone responsible for analytical methods development; the supervisor reports to the ESE Project Manager and the USATHAMA Central Laboratory QA Coordinator. The specific responsibilities of the QA/QC Supervisor are detailed in Paragraph 2.2.2.

The analyst, under the supervision of the Analytical Team Leader, performs the analyses associated with methods development and submits the results to the Analytical Team Leader for approval. The Analytical Team Leader writes the methods development reports in approved USATHAMA format. The Analytical Team Leader also writes the Proposed Method Development Plan in the USATHAMA format prior to the beginning of work on a method. The Method Development Plan will be submitted to USATHAMA for approval before work begins.



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The personnel having a direct role in project QA/QC are the ESE Project QA/QC Supervisor, the USATHAMA Central Laboratory QA Coordinator, the ESE Project Manager, the ESE Analytical Team Leader, and the analysts.

2.2 QA/QC RESPONSIBILITIES

2.2.1 USATHAMA CENTRAL LABORATORY QA COORDINATOR

The Central Laboratory of USATHAMA is responsible to the Commander of the Agency for the quality of data collected in support of its programs. The USATHAMA Central Laboratory QA Coordinator, therefore, has the following responsibilities in fulfilling this objective:

- 1. To provide technical evaluation of field laboratory QA/QC procedures;
- 2. To provide Standard Analytical Reference Materials (SARMS) with supporting documentation to field laboratories;
- 3. To provide QA training as required;
- To provide technical evaluation of field laboratory methods development documentation;
- 5. To notify the USATHAMA Project Officer, ESE Project Director, ESE Project Manager, and ESE QA/QC Supervisor when a situation exists which precludes statistical control of results; and
- t. To provide continuous review of the implementation of the field laboratory QA/QC plan and report the findings to the USATHAMA Project Officer.

2.2.2 ESE PROJECT QA/QC SUPERVISOR

The ESE Project QA/QC Supervisor is responsible to the ESE Project Manager and the USATHAMA Central Laboratory QA Coordinator to monitor the quality of all data reported to USATHAMA in the method report and documentation. The supervisor's specific responsibilities are:

1. To provide an independent overview of the QC practices of the project team from beginning of the project through acceptance of the final report;

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- 2. To ensure that the team completes all QC requirements of the project plan;
- 3. To approve each method report in the USATHAMA format, and to ensure that the documentation data are correct;
- 4. To audit data files for correct entry of all data;
- 5. To assure the availability of SARMS or, where unavailable, to approve the use of interim reference materials;
- 6. To arrange for and report on purity analyses and stability checks on interim reference materials;
- 7. To assure the delivery of interim reference materials to the Central QC Laboratory;
- 8. To establish and maintain liaison between the ESE Project Team and the USATHAMA Central QA Coordinator; and
- 9. To maintain a vigil of the entire laboratory in order to detect conditions which might jeopardize control of the various analytical systems.

2 1. 3 SSE PROJECT MANAGER

The ESR Project Manager is responsible for effective day-to-day can exemple of the total project staff, as well as direct communication and liaison with the USATHAMA Project Officer. The Project Manager's responsibility specific to QA/QC is to approve all QA/QC procedures to be used in the conduct of the project, to provide additional authority when required to support the ESE Project QA/QC Supervisor, and to approve of any revisions to the project QC plan.

The Project Manager is responsible for effective day-to-day coordination of all USATHAMA activity, guidance and technical support in resolution of QC problems, and support of QA/QC preparation of unknown reference samples. This manager also provides additional authority, when needed, to support the QA/QC Supervisor in analytical matters.

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2.2.4 ESE ANALYTICAL LEADER

The ESE Analytical Leader is responsible for provision of accurate laboratory data produced by analysts under his supervision. He is responsible to the ESE Project QA/QC Supervisor to ensure that all quality control procedures are followed and documentation provided. The QA role of the Analytical Leader is, therefore, to assist the QA supervisor in enforcing QA/QC procedures.

2.2.5 ESE ANALYSTS AND SAMPLING PERSONNEL

The following sections describe the QA/QC procedures required to define and document the validity of data forthcoming from the conduct of this project. It is the responsibility of the analysts to perform the required QA/QC procedures and to document all observations in logbooks in permanent ink. It is also the responsibility of the analyst to perform preliminary QC checks to ensure that each batch of data being generated meets all analytical systems criteria. The analyst must also bring any unusual observation or analytical problem to the immediate attention of his/her Analytical Leader or the ESE Project QA/QC Supervisor.

Each analyst is responsible for ensuring that sufficient quantities of reagents of adequate quality are available for the performance of the required analyses.

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3.0 DOCUMENT CONTROL AND REVISIONS

3.1 RATIONALE

The Project Quality Assurance Plan includes a system for documenting and updating all sampling, analytical, and data handling procedures used by ESE for this project. This system uses a standardized indexing format and provides for easy replacement of pages, if techniques and procedures are changed.

3.2 STANDARD OPERATING PROCEDURES

The standardized indexing format includes, at the top of each page, the following information:

- 1. Section Number,
- 2. Revision Number, and
- 3. Date of Revision.

Section numbers within a document are in numerical order. Revision number represents the most current version of a section, with the original version listed as "O". The date represents the date of the latest version. The text of each major section begins on a new page. If revisions to a section involve expansion which adds pages, the additional pages will be numbered la, lb, lc, etc. For example, if Page 4 were revised and expanded to include an extra paragraph, the overflow would appear on a page designated 4a. The original Page 4 would then be removed from the manual and replaced by revised Page 4 and Page 4a. This system allows expansion within a section without renumbering the entire document.

The Table of Contents follows the same structure as the text and contains a space for "Revision." When a revision to the text is made,

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the Table of Contents page is updated by retyping, or by striking out the old revision number and printing in the current revision number.

Proposed revisions will be reviewed by the Project QA/QC Supervisor, Project Manager, and Project Director and then sent to the USATHAMA Central QA Laboratory Coordinator and USATHAMA Project Officer for final approval. AMD. 1/QC4.1

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4.0 ANALYST TRAINING AND CERTIFICATION

4.1 RATIONALE

Accurate and precise analyses can be conducted only by well-trained analysts who correctly operate instruments, thoroughly understand analytical methods, use good analytical technique, and understand and practice necessary QC procedures. While the necessary training may initially be obtained from education, experience, or on-the-job training, it is imperative that the analyst's capabilities be verified prior to conducting analyses and reviewed periodically thereafter.

4.2 STANDARD OPERATING PROCEDURES

The QA/QC Supervisor will provide "test" samples and assist ESE Technical Operations Management when possible in the training of sampling team members and analysts; however, such training is the ultimate responsibility of ESE Technical Operations Management.

Direct responsibility for analytical training rests with the ESE administrative management level of Group Leader.

Each analyst will be reviewed by his/her Group Leader to ensure:

- 1. Working knowledge of QC policies,
- 2. Preparation of standards and spikes,
- 3. Acceptable analysis of reference samples,
- 4. Acceptable analysis of replicates and spikes.
- 5. Detection limit and standard calibration requirements, and
- 6. Knowledge of preventive maintenance techniques.

This evaluation must be done for every analysis the analyst performs on this project.

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A list of qualified personnel for each sampling and analytical task will be provided by the appropriate Group Leaders to the ESE QA/QC Supervisor.

The QA/QC Supervisor will keep a logbook arranged by type of analysis (e.g., Autoanalyzer, atomic absorption, gas chromatography, GC/MS, etc.). Analysts' names will be entered under the qualified headings with the Group Leader's initials and date certified (Figure 4-1). At annual intervals, each Group Leader will review the capabilities of each analyst and recommend whether certification should be continued.

4.3 PERFORMANCE AUDIT

During the conduct of this project, the QA/QC Supervisor will inspect the laboratory occasionally to determine if analyses are being performed only by certified analysts. Data reports require the name of the analyst on the report sheet. Reruns of samples may be required if certified analysts did not perform the analysis.

4.4 ANALYST CERTIFICATION PROCEDURES

The confidence in any analytical method is limited if the analyst has not demonstrated skill in performing the analysis. Analysts will, therefore, not only be trained in QC techniques, but also be required to qualify to run analyses. The qualification test results for certifying analysts must be statistically valid and must include evaluation of precision and accuracy.

Analysts will demonstrate their proficiency in conducting chemical analyses by analysing spiked standard samples using approved analytical methods. Proficiency will be demonstrated for each analyte to be analyzed by the analyst prior to conducting analyses of natural samples.

For any analytical method, analysts or an analytical team consisting of specific individuals will be considered to be certified to run a particular analysis if they have been involved in developing the precision

AMALYSIS TYPE AUDITOR Certified Yes | No Analyst Dete Coument Analysis SCURCE: End Prepared for: U.S. Army Toxic and Hazardous Materials Agency Figure 41

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and accuracy data needed for method documentation. The precision and accuracy data generated during method documentation must be acceptable to the Analytical Team Leader and the QA/QC Supervisor.

The analytical team responsible for gas chromatographic (GC) and highperformance liquid chromatographic (HPLC) analyses will usually consist
of a technician performing the sample work-up and extraction and a
chemist performing the subsequent instrumental analysis of the extract.
Should this team fail to pass the required QC tests of precision and
accuracy for a certain batch of samples, corrective action will require
the investigation of the instrumental analysis separate from the
extraction. The chemist performing the instrumental analysis is
considered certified if he/she meets the instrumental calibration QC
criteria. The extraction procedure will then be identified as out of
control. The extraction technician can gain recertification by
repeating the extraction in question and meeting the QC criteria. If
the analyst fails to produce acceptable results, analysis is stopped
until the reason for failure is identified and corrected.

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5.0 LABORATORY AND METHOD CERTIFICATION

5.1 RATIONALE

Each new method developed requires documentation including precision and accuracy data. A specified detection limit must be achievable for a particular analyte. This detection limit must be statistically meaningful and consistently obtainable. This section outlines the testing procedures which will be used to define the detection limit, precision, and accuracy of each analytical method, according to standardized criteria.

5.2 METHOD CERTIFICATION

The following paragraphs describe the procedures to be used to certify analytical methods. All methods certification and documentation data will be developed initially in standard matrices and subsequently in natural samples.

The standard matrix for documentation of organic analysis will be deionized, organic-free (ASTM Type IV) water containing 100 milligrams per liter (mg/L) each of sulfate and chloride prepared as follows:

- Add 1.48 grams (g) of dried reagent-grade anhydrous sodium sulfate to a 1-liter (L) volumetric flask and dilute to volume;
- 2. Add 1.65 g of dried reagent grade sodium chloride to a 1-L volumetric flask and dilute to volume; and
- 3. Transfer 100 milliliters (ml) of each (1 and 2) to a 1-L flask and dilute to volume.

The resulting solution contains 100 mg/L each of chloride and sulfate ions. This water will be used for blanks or will be spiked with the compound(s) of interest prior to processing through the complete analytical protocol.

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The natural water sample used in method documentation can be any uncontaminated natural water sample, preferably a surface water or a composite of surface and ground water, which is available in sufficient quantities to conduct the natural sample testing for all the analytical methods developed under this contract. The same natural water should be used for all analytical methods documentation.

The data for documentation of analyses in soils will be developed using a standard soil matrix. The standard soil will consist of a homogeneous sample of sufficient size to provide a single continuous source for all method documentation. An aliquot of sieved (Paragraph 7.1.3.2) standard soil will be carried through each set of documentation samples to act as a blank. Added concentrations of the subject analyte(s) will be dissolved in a volume of solvent just sufficient to wet the soil. This solution is poured over the subsample of soil and allowed to stand for 1 hour prior to beginning analysis.

The natural soil sample to be used in method documentation can be any uncontaminated soil, preferably a composite of several soil types, which is dried, sieved, and available in sufficient quantities for all the method documentation work to be performed in this project.

Quantitative analytical methods will be developed in this project to quantitate the level of contamination of specific analytes in various environmental matrices. The process of quantitative documentation requires the detection limit of the analytical method to be determined and the precision and accuracy documented to a statistically reliable degree.

Certification of a quantitative method requires analysis of separate batches of standard media (water, soil, etc.) samples spiked at the following concentrations: 0.5%, 1.0%, 2.0%, 5.0%, and 10%, where % is the required/desired detection limit. Each level is spiked and analysed once on 4 separate days. A blank sample of the standard matrix is also analysed with each batch.

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A plot of found-versus-target concentration (amount spiked) is generated, and the detection limit is calculated using the methods of Hubaux and Vos. The precision of the method will be the standard error of the best-fit linear regression line of found-versus-target concentration values for the data generated over the 4 days of certification testing. The accuracy of the method will be the slope of the best-fit linear regression line of found-versus-target concentrations. The method will be written in the USATHAMA format and will be submitted to USATHAMA for approval along with the certification data.

6.0 SAMPLE COLLECTION

6.1 RATIONALE

As part of the verification of the developed methods in natural water/soil samples, it may be necessary to collect contaminated or "blank" natural samples for analysis by the methods developed during the project.

6.2 QA PROCEDURES FOR SAMPLING

A field team and leader will be designated by the Project Manager. The Field Team Leader is responsible for proper sampling, labeling of samples, preservation, and shipment of samples to the laboratory in a proper manner.

Water samples will be stored and shipped in amber glass bottles with Teflon—lined lids. Soil samples will be contained in glass Mason jars. These containers will be prepared by thorough washing with hot detergent and water, triple rinsing with tap water, triple rinsing with deionized water, rinsing with methanol, air drying, and baking at 100°C for several hours.

Sampling locations will be determined in discussions between the Project Manager and the USATHAMA Project Officer.

All collected samples will be stored under refrigeration at 4°C at all times prior to subsampling or analysis.

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7.0 ANALYTICAL SYSTEMS CONTROL

7.1 GENERAL PROCEDURES

7.1.1 NOTEBOOKS AND INSTRUMENT LOGBOOKS

The ultimate repository for information concerning analyses performed in the laboratory is the analyst's personal laboratory notebook and the instrument logbooks.

Each analyst is required to have a personal notebook which is designated by a unique number. Each analyst is responsible for maintaining complete laboratory notes. The ESE QA/QC Supervisor maintains a list of assigned notebook numbers and audits laboratory notebooks without notice. The list of assigned notebooks contains the following information:

- 1. Notebook number,
- 2. Assignee,
- 3. Responsible Group Leader, and
- 4. Disposition or location and date.

Laboratory notebooks for this project will not be taken from ESE without written permission of the Analytical Team Leader and the ESE Project Manager. After completed notebooks are approved by the analyst, QA/QC Supervisor, and Group Leader, custody is transferred to the Analytical Team Leader. Every entry into the notebook should be dated and signed. Each Group Leader is responsible for ensuring that the notebook entries are counter-signed. Entries in the personal notebook will vary depending on the role of the individual in the laboratory and the type of work being performed. At a minimum, the personal notebook should contain:

 A reference to or a description of the procedures used for sample work-up or analysis,

- 2. A summary of the samples extracted or analyzed,
- 3. Weighings and calculations of standard concentrations, and
- 4. Information on spiking procedures, and observations and comments on the procedures or samples.

The logbook consists of a bound notebook containing the preventive maintenance schedule.

Each time an instrument is used for sample analysis, the following information is entered:

- 1. Date of analysis;
- 2. Project name and number;
- 3. Number of samples analyzed, and type of sample;
- 4. Time spent on analysis (start to finish);
- 5. Preventive maintenance performed, if any;
- 6. Time spent on preventive maintenance;
- 7. Instrument calibration performed, if any; and
- 8. Name of analyst.

Additional notes are made in the instrument logs when required. These notes are particularly important when abnormal instrument or analytical performance is observed. It is the analyst's responsibility to ensure that instrument logs are properly filled out and kept up to date. The QA/QC Supervisor monitors and audits the status of instrument logbooks.

Copies of all pages of instrument logbooks for instruments used in this project will be compiled monthly by the analyst and approved by the QA/QC Supervisor. This instrument documentation record becomes a part of the permanent project QA/QC record.

7.1.2 STANDARDS AND SARMS

All materials used to prepare calibration standards and spiking standards must be Standard Analytical Reference Materials (SARMS)

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supplied by USATHAMA. SARMS or interim SARMS are materials that have undergone extensive purity and stability checks. Interim reference materials may be used when analyses must be run before a SARM is available. However, the following precautions must be taken:

- 1. The interim reference material will be stored at 0°C, and a portion will be retained for comparison with the approved SARM when available;
- 2. The following data will be recorded as a minimum description of the material:
 - a. Infrared spectrum;
 - b. Melting point, decomposition point, or boiling point;
 - c. NMR spectrum;
 - d. Elemental analysis; and
 - e. GC or LC (by difference) analysis.

In cases where SARMS are difficult to obtain or only small amounts are available, interim SARMS or standards may be used for all calibration and spiking work, as long as the purity and response of such materials can be compared to the purity of the SARM. All reference compounds used in this project will be stored at 0°C and protected from light. The QA/QC Supervisor or Analytical Team Leader will request SARMS as required. The QA/QC Supervisor maintains a record of receipt of SARMS and monitors their use. A record of SARM material usage is maintained which identifies the analyst and date of use.

7.1.3 SAMPLE PREPARATION

The following paragraphs describe the preparation of water, soil, and sediment samples for analysis.

7.1.3.1 Water Samples

Prior to analysis, groundwater samples will be filtered through a 0.45-micron filter (constructed of a material which is suitable for the intended analysis) to remove suspended particulate matter. Surface water samples will not be filtered before analysis. Many organic

compounds adsorb on particulate matter. Therefore, it is desirable to detect contaminants migrating in the suspended fraction of the water column.

7.1.3.2 Soil Samples

Prior to analysis, soil samples will be sieved through a 30-mesh (500-um) brass sieve to remove rocks and debris.

Prior to sieving, soil samples should be spread out on the dull side of aluminum foil to air dry if sufficiently wet.

Soil samples must be properly subsampled before any analysis is performed. All subsampling must be accomplished with the aid of a riffle or by proper quartering techniques according to ASTM Specification D346.

A moisture determination in accordance with ASTM Specification D2216-71, Laboratory Determination of Moisture Content of Soil, will be made on each solid sample so that analytical data can be reported on a moisture-free basis.

7.1.4 STANDARD SAMPLES

Preparation of standard water and soil for methods development and analytical systems control have been described in Paragraph 5.2. Standard water for inorganic analysis consists of deionized water. Standard water for organic analysis consists of deionized organic-free water containing 100 mg/L each of sulfate and chloride. Standard samples for soil and sediment analysis consist of samples of an approved standard soil.

7.1.5 INSTRUMENT CALIBRATION AND OPERATING PROCEDURES

A calibration procedure establishes the relationship between an accurately known calibration standard and the measurement of that

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standard by an instrument or analytical procedure. Calibration is not to be confused with standardization. Standards are run each time an instrument or procedure is used, while calibration is performed only at specified intervals.

Operating procedures must be available for all equipment and analytical instrumentation. Such procedures are generally provided by the manufacturer.

Written procedures for the operation and calibration of instrumentation are provided to the analyst in the laboratory to help minimize possible measurement inconsistencies due to differing techniques, conditions, and choice of standards. The procedures include the following information:

- 1. The specific instrument (or group of instruments) and analysis for which the procedure is applicable;
- An explanation of theoretical considerations pertinent to the understanding of both the calibration procedure and the analysis;
- 3. Fundamental calibration specifications;
- 4. A list of requisite standards and equipment for the procedure;
- 5. Complete presentation of the procedure in a clear, step-by-step manner; and
- 6. Specific instructions for obtaining and recording calibration information.

An up-to-date report for each calibration standard used in the calibration system should be provided. If calibration services are performed by a commercial laboratory on a contract basis, copies of reports issued by them should be maintained on file.

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All contractor calibration reports are kept in a suitable file by the QA Supervisor and contain the following information:

- 1. Report number;
- Identification or serial number of the calibration standard to which the report pertains;
- Conditions under which the calibration was performed (temperature, relative humidity, etc.);
- Accuracy of calibration standards (expressed in percentage or other suitable terms);
- 5. Deviation or corrections; and
- 6. Corrections that must be applied if standard conditions of temperature, etc., are not met or differ from those at place of calibration.

Contracts for calibration services should require the contractor to supply records on traceability of their calibration standards.

All equipment to be calibrated should have affixed to it in plain sight a tag bearing the following information:

Description:	
Ident. No.:	 _
Last Calibrated:	
Calibrated By:	
Calibration Expires:	

NOTE: Use of this instrument beyond the calibration expiration date is prohibited.

When the equipment size or its intended use limits the application of labels, an identifying code should be applied.

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Equipment past due for calibration should be removed from service either physically or, if this is impractical, impounded by tagging or other means.

7.2 SPECIFIC ANALYTICAL SYSTEMS CONTROL: GC AND HPLC ANALYSES

7.2.1 INSTRUMENT CALIBRATION

Before analyses are performed on the gas chromatograph, the gas flow rates through the column and detector are measured and the carrier gas flow controllers are calibrated. For roto-meter controllers, the position of the floating ball is measured versus the gas flow through the column and a graph of flow rate versus ball position is drawn. Electronic flow controllers are calibrated according to manufacturer's specifications, and the agreement between the set flow rate and the measured flow through the column is checked. A table of measured versus set flow rate is prepared. The flow rate through the column and detectors is measured using a volumetric bubblemeter supplied by the manufacturer. A packed column should be in the instrument, and the oven and the injector should be at ambient temperature. The flow-rate setting is checked at the start of an analytical run and periodically calibrated thereafter, but at least once every 6 months. A record of the flow rate calibration including charts and tables is kept in the instrument logbook.

Temperature calibration of the detector, injector, and oven zones in a GC is accomplished before any analyses are performed. The temperature in these zones is measured by a calibrated thermocouple placed into the appropriate zone. The temperature calibration is recorded in the instrument logbook.

For high pressure liquid chromatographs, the system flow rates are calibrated before any analyses are conducted. The calibration is performed by measuring the liquid flow at the outlet of the detector system using a volumetric measurement such as a Class A volumetric flask. In two pump gradient systems, the flow rates should be checked

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with each pump working independently at a 50/50 gradient mixture. A record of this calibration should be kept in the instrument log book.

HPLC columns should be checked for efficiency, before any analysis is conducted, according to manufacturer's specifications or another approved consistent procedure. A record of the column's efficiency and resolution between adjacent peaks should be kept in the instrument notebook.

Calibration is performed when a new instrument arrives in the laboratory and is repeated at periodic intervals thereafter. The instrument sensitivity calibration is performed according to the respective manufacturer's instructions. This sensitivity or performance check is repeated when instrument performance deterioration is suspected. When no manufacturer-supplied sensitivity check procedures are available, the instrument response to a known concentration standard under defined conditions is to be used to check the sensitivity.

The instrument logbooks should contain:

- Data of last calibration of flow and temperature controllers, and historical curves,
- 2. Detector calibration calculations and information,
- 3. A log of the type of analysis run on the instrument including:
 - a. Column conditions and temperature zones for GC and column type, conditions and flow rates for HPLC.
 - Sample numbers or other identification of samples analyzed.
 - c. Reference to a notebook page describing the analysis performed.
 - d. Date of analysis.
 - e. Detector used--FID, ECD, UV, fluorescence, etc.
- 4. Service records.

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7.2.2 PEAK MEASUREMENTS

Three primary means of measuring chromatographic peak response may be used: area, peak height, and peak height x width-at-half-height. Peak height is preferred in those cases where very noisy signals make the use of electronic integrators difficult in complex chromatograms. In this project, all these means of peak response measurement may be used. However, once the choice of peak response measurement is made, the same mode of peak response measurement will be used consistently throughout the project for that particular analysis. The choice of method used for a particular analysis is made by the analyst after consultation with his/her supervisor.

Analyte peaks on a chromatogram must be identified. The peaks of interest must be marked to definitively specify which peaks were used in the quantitation and calculations.

The primary means of peak identification for an analyte in the chromatographic methods is that the peak of interest falls within a predetermined retention time window of the retention time of an authentic standard of that particular analyte. Usually a retention window of 5% will be used for peak identification. However, for temperature-programmed or solvent-programmed runs, a retention window of ±2 standard deviation units around the average value of the standard peak retention time should be used as a confirmatory check. The average peak retention time is determined from analytical standards run that day.

7.2.3 SAMPLE DOCUMENTATION

An analyst's extraction logsheet will be filled out by the analyst performing the sample extraction and will accompany the batch of samples throughout the analysis procedure. (An example of this extraction logsheet is given in Figure 7-1). The data on this sheet will include, at a minimum, the following:

USATHAMA.2/EXTRACT	-LOG.1							
Project Name:					¹	Logbook N	b.:	
Project No.:						Plant Cod	•: <u> </u>	
						Analyst:	-	
Analysis:								
Extraction Procedu	re:							
								·
		pii Adjusted	Final Extract	if	Initial Dry Weight if	Detrac-		·
Sample Number Date	Sample (1) (kg)		Volume (mi)	Applic- able	Applic- able	tion Solvent	Final Solvent	Comments
	<u> </u>					<u> </u>	<u> </u>	
[
			-					
								
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	-							
						-		
	<u> </u>		L	L				es and Barbaratan by some
22						pered f		see and Engineering, Inc., 1990.
Figure 7-1 ESE EXTRACTION	on log	SHEET			U.S. Met	. Army erials /	Toxic Agenc	and Hezerdous y round, Meryland

- Notebook or literature reference to procedure used for extraction,
- 2. Type of sample matrix,
- 3. Date of extraction,
- 4. Volume or weight of sample taken for analysis,
- 5. Dry-weight of the sample-if necessary,
- 6. Final volume of extract,
- 7. Project number and name,
- 8. Notes and comments affecting the extraction procedure, and
- 9. Name or initials of the analyst.

7.2.4 CHROMATOGRAPHIC LOGSHEETS

Two types of chromatographic logsheets are used. One sheet is the standard curve sheet (Figure 7-2) which lists the standards, their concentrations, and the respective peak heights or areas. The second sheet, the chromatographic data sheet (Figure 7-3), lists the samples in order of their injection with the factors needed for calculation of the concentrations.

7.3 ANALYTICAL SYSTEMS QUALITY CONTROL PROCEDURES

The following describes the quality control procedures and requirements for analyses conducted during this project. These quality control requirements are in addition to any specific calibration requirements presented in Subsection 7.2.

An initial instrumental standardization will be performed before any samples are analyzed. Calibration standards will be prepared and analyzed in the concentration series 0 (blank), 0.5%, X, 2%, 5%, and 10% where X is the concentration in the extract or sample being analyzed corresponding to the desired detection limit. For example, if the desired detection limit in the matrix is 1 microgram per liter (ug/L) and a thousandfold concentration is required before introduction into an instrument, X would be 1 ug/L. The data from the initial calibrations during documentation will be averaged and used to calculate: (1) the

Chromatogram Mumber Prepared for: U.S. Army Toxic and Hazardous Curve No. 3 Aberdeen Proving Ground, Maryland Page: Normalized Response Attn/Pactor Curve No. 2 Materials Agency Compound: Retention Time: Date: Analyst: Logbook No.: Area Peak or Height POUNCE: Ends Curve No. 1 STANDARD CURVE DATA SHERT FOR CC/MPLC AMALYSES ENVINOMENTAL SCIENCE AND ENGINEERING, INC. ALE. Stds.. Used Corr. Coef. Intercept Slope įż Conc. Normalized Response Attn/Pector Figure 7 - 2 Ede Standard Curve Data sheet for BC/HPLC Analyses Curve No. 3 Area Peak or Reight Att. Curve No. 2 įi įį Curve No. 1 QABIVA. 3/BATA-STUNKVE. 1 Project: Project Musher: Plant/10: ij Compound: Return ion Time: Stds. Bead Corr. Coef. Intercept Slope ž : • -2 • •

i

SOUNCE: Endommental Science and Engineering, Inc., 1988. Chromat og ram Number Prepared for: U.S. Army Toxic and Hazardous Materials Agency Std. Curve Used Sample Concen-tration Date: Initial Sample Wr. or Vol. ENVIRONMENTAL SCIENCE AND ENCINEERING, INC. Amount In j. Ext CHROMATOGRAPHIC LOG SHEET SAMPLE ND. Pages: Amount Ext (ug) Dilu-tion Ext Vol Normalized Response Attn/ Factor Log Book No.: Peak Height or Area Figure 7 - 3 ESE CHROMATOGRAPHIC LOGSHEET Acc letention Time Mindow (Z) In j Vol QADIV4. 3/CHRONA-LUG. 1,2 Ĵz P J Project: Project No.: Analyst: Sample Type: Compound

Aberdeen Proving Ground, Maryland

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average instrumental Hubaux and Vos detection limit, and (2) the average slope of the calibration curve.

The average slope will be used to initiate and set up the control limits. During the development of the method certification data for each analyte, each instrument will be controlled using a standard curve consisting of three standards plus one procedure blank analyzed at the beginning of the analytical batch. A single standard (mid-scale) will be analyzed at the end of the analysis to measure instrument drift. An instrument shall be considered out of control if the slope of the calibration curve decreases by more than 30 percent. The response values for the mid-scale standard analyzed before and after the run must agree within 30 percent. The correlation coefficient for the linear regression of the calibration curve must always exceed 0.995. The value of each of these checks will be recorded in the analyst's notebook.

The analysts are responsible for ensuring that each routine standardization meets the QC criteria. Failure of the analytical system to meet all QC criteria requires immediate corrective action, which may include rerunning samples judged out of control.

All data reported using the calibration curve must be bracketed on the upper and lower end by standards.

At least one procedure blank sample will be included with each batch of samples.

7.4 DEVELOPMENT OF ANALYTICAL METHODS

Development of a method will be initiated by submitting Documentation for Proposed Methods Development to the USATHAMA Chemistry Group for approval prior to development activity (Appendix 4 of USATHAMA QA Program, 1980). Each of the methods proposals will contain the following information, organized in the format outlined below:

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DOCUMENTATION FOR PROPOSED METHOD DEVELOPMENT: (ANALYTE) IN (SOIL/WATER)

- 1. SUBMITTING ORGANIZATION.
- 2. JUSTIFICATION OF WORK.
- 3. OUTLINE OF PROPOSED EFFORT.
- 4. PROPOSED SAMPLES TO BE USED IN DEVELOPMENT.
- 5. ESTIMATE OF RESOURCES REQUIRED.
- 6. PROPOSED SOURCE OF RESOURCES.
- 7. IMPACT OF REPROGRAMMING USATHAMA FUNDS.
- 8. ESTIMATED TIME TO COMPLETE WORK AND SUBMIT REPORT.

The USATHAMA Chemistry Group will evaluate the proposed approach for technical soundness and economy of effort. The Chemistry Group will then request that ESE proceed with the method development as proposed or with recommended modifications.

ESE personnel will investigate the proposed procedures to be included in the method. Should any of the proposed procedures approved by the Chemistry Group be found to be inadequate for the method, alternative procedures will be investigated after approval by the Chemistry Group.

7.5 CHARACTERIZATION OF ANALYTICAL METHODS

When the analytical procedures have been finalized by ESE, the procedures will be documented according to the requirements specified in Appendix 2 of the USATHAMA QA Program (August 1980).

ESE will generate precision and accuracy data on the proposed method in standard samples and in natural media.

Using the precision and accuracy data, the detection limit will be calculated as well as the sensitivity at the detection limit for inclusion in the documentation of the method.

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Full documentation of the method will be submitted to the USATHAMA Chemistry Group. The Chemistry Group will review the documentation for completeness and comprehension. Based on this review, the Development Laboratory will make any necessary modifications prior to approval of the method by the USATHAMA Chemistry Group.

These method documentation data will include estimates of the standard deviation, percent inaccuracy, and percent imprecision.

1. The standard deviations will be calculated at each target concentration according to:

Standard deviation =
$$S = \left[\sum_{i=1}^{n} \frac{(\sum_{i=1}^{n})^2}{n-1} \right]^{1/2}$$

where X_i = the ith found concentration

n = total number of X values

and Σ = summation from i - i to i = n

2. The percent inaccuracy will be calculated at each target concentration according to

percent inaccuracy =
$$\frac{x - TC}{TC} \times 100$$

where x = average found concentration at the particular TC and TC = target concentration.

 The percent imprecision will be calculated at each target concentration according to

percent imprecision = $\frac{s}{x} \times 100$

where s = standard deviation

and $\overline{\mathbf{x}}$ = average found concentration at the particular target concentration.

Upon final approval of the documented method, the Chemistry Group will assign a number to the method.

The format below (USATHAMA QA Program, August 1980) will be followed for submittal of all method documentation data.

TITLE: ANALYTE(S) IN (WATER/SOIL) SAMPLES

- 1. Application: State analytes that can be analyzed by this method and media in which the analytes are contained (e.g., TNT in soil). The media should be the original matrices to be analyzed (e.g., soil, air, water, biological tissue, etc.) rather than intermediates in the procedure (e.g., bubbler from air sampling, extract from soil, etc.).
 - a. Tested Concentration Range: State concentration range in the original matrix that was tested for this validation (e.g., 1 to 20 ug/L in water, 5 to 100 mg/m³ in air, etc.).
 - b. <u>Sensitivity</u>: Response (peak height, area, etc.) observed for absolute quantity of analyte (state quantity) at the detection limit (e.g., 1,500 area units for 40 picograms).
 - c. Detection Limit: Limit of detection for complete analytical method, determined from precision and accuracy data generated from spiked standard samples (standard water, soil, etc.) and calculated according to Hubaux and Vos, expressed in terms of concentration in original medium (soil, water, etc.).
 - d. <u>Interferences</u>: State any observed interferences or any interferences anticipated based on the method of analysis.
 - e. Analysis Rate: State the estimated number of samples that can be analyzed by this method in an 8-hour day after instrument calibration.

2. Chemistry:

- a. List physical and chemical properties of analyte(s), including Chemical Abstracts Service Registry number.
- b. Describe in detail any chemical reactions involved in the analytical method (such as conversion of organic nitrogen to ammonia followed by conversion to ammonium chloride in hydrochloric acid).

3. Apparatus:

- a. <u>Instrumentation</u>: List makes and models of instruments, as well as specific characteristics (such as detectors).
- b. <u>Parameters</u>: List operating parameters of instruments, as well as chromatography columns.
- c. <u>Hardware/Glassware</u>: List miscellaneous equipment. Include sources for specialty or trademarked items.
- d. <u>Chemicals</u>: List chemicals necessary. State sources of analytical reference materials.

4. Standards:

- a. <u>Calibration standards</u>: Describe, in detail, the step-bystep procedure for preparing instrument calibration standards to include proper storage and shelf life.
- b. <u>Control spikes</u>: Describe, in detail, the step-by-step procedure for preparing spikes of control samples.
- 5. Procedure: Describe, in detail, the step-by-step procedure for analyzing control and actual samples, as well as instrument calibration procedures. Include instructions for constructing necessary graphs.
- 6. <u>Calculations</u>: Describe, in detail, the manner by which the concentrations in the original matrix are calculated from the responses obtained in the analysis.

7. References: List any references used as a source for the procedures.

8. Data:

- a. Tabulate precision and accuracy data by indicating found concentrations (uncorrected) for each target concentration by day.
- b. Tabulate average found value, standard deviation, percent imprecision, and percent inaccuracy for each target value.
- c. Plot the found concentration versus the target concentration (include linear regression, confidence bounds, and Hubaux and Vos detection limit annoted).
- d. Plot the standard deviation versus the target concentration.
- e. Plot the percent inaccuracy versus the target concentration.
- f. Plot the percent imprecision versus the target concentration.

APPENDIX C
COMPUTER PROGRAM FOR REDUCTION
OF HPLC SCREEN DATA

APPENDIX C

COMPUTER PROGRAM FOR REDUCTION OF HPLC SCREEN DATA

The various subsections of the program are described below:

Steps 10 through 300—Calculates the ug/L of the analytes in the original sample from the raw data and stores the peak heights for later calculation of ratios.

Steps 400 through 430~Lists the analyte names under the file variables H1(I) and H2(I) which are the first and last parts of the compound name, respectively.

Steps 600 through 710—Lists a set of calibration curve data for an analyte on a particular detector.

Steps 992 through 1446—Calculates the calibration data for all of the analytes and stores the resultant slope, intercept, and correlation coefficient for subsequent use under a compound index number, I, and a file number, FI, for each detector:

FI = 1 for the LC-75

FI = 2 for the 254 nm

FI = 3 for fluorescence

The compound index number, I, is equivalent to the X index number used in the calibration curve data. The correlation coefficient is stored under Gu(X). The slope and intercept are stored under RS(X) and RC(X), respectively.

Steps 1495 through 1860—Calculates the detector ratios for each analyte in a single chromatographic run. B1(I), B2(I), and B3(I) are the peak heights of component I on the LC-75, 254 nm, and

fluorescence detectors, respectively. The ratio of the peak height of component I on the LC-75 detector to the peak height on the 254 nm detector is stored as T(I) for responses normalized to the ortho-nitrotoluene response and V(I) for the unnormalized response. H(I) and S(I) are used for the unnormalized 254 nm to fluorescence ratio and LC-75 to fluorescence ratio, respectively.

Steps 1899 through 1932--Subroutine for manual entry of peak heights for the 254 nm detector.

Steps 1969 to 1998--Manual entry of peak heights for fluorescence detector. (Manual entry for LC-75 peak heights uses Steps 130 to 150.)

Step 2082--Defines a special function key for initiation of the ratio program at 992.

Step 2086--Defines a special function key for initiation of the data reduction program at Step 15.

An example of the output of the ratio calculation program beginning at Step 1495 is presented below. Ratios unnormalized and normalized responses are calculated.

Step 2090--Defines a special function key for initiation of the detector ratio program at 1495.

The following is a listing of the input and output of a typical data set calculation for the LC-75 data. In the input section, the program provides a series of prompting statements to set up the data file, and the peak heights are entered for each prompted compound. The output section is titled report and contains the concentration in the sample, the peak height as a check for input errors, and the ratio of the peak height to the peak height of ortho-nitrotoluene.

```
RUN 1495
WHAT LEVEL
7 19
```

EHTER	OHT	RESPONSE	AT	LC-25
2 519	0 W T	RESPONSE		254
2 440	7741	*63-0436	~'	27408

PERPANSE	RATIOS	t	LEVEL	18.
***	ROTTA	WITH	OHTees	

COMPOUND	LO-25/254
H#X	7.1311492
PDX	3, 1915717
THE	3, 9388281
DNS :	2, 8388309
TETRVL	A.
DHP	1, 4372199

THT	2.578323
2,6 DHT	2.068782
2, 4 BHT	2.4518561
ONT	1.
•	
HRPHT	1.535228
ACENAPHTHY	1, 4738333
FLUORENE	8. 1774753
-ACEHAPTH	9. 5486892
HEHANTH	0.401843
ANTHRACENE	0.8878672
FLUORANTH	2.6348618
PYRENE	0.5352684
CHRYSEHE	4. 4791968
SZ(B)FLUOR	1. 12634
BZ(K)FLUOR	1.383748
B(a)PYREHE	1.7397887
DBZ(AH)AHT	13.643839
I CCD>PYR	1.0171319
4 / W W " 1 / 7 / 7	* * * * * * * * * * * * * * * * * * *

PATIOS WITHOUT ONT

COMPOUND	LC-75/254	254/FL	LC-75/FL
4#X	A. 5834872	A.	●.
PDX	2. 9815838	●.	0.
THE	3, 592093	•.	●.
DMO	1.8593564	A,	i.
TETRYL	0.	9.	₿.
DHP	1.318784	0.	Ö.
THT	2. 3440621	•.	0.
2, 6 BHT	1. 8865979	ė.	Ö.
2.4 BHT	2. 2352941	0.	Ö.
ONT	9. 9119748	ē.	Ĭ,
HRPHT	1.4888847		37. 579545
ACEHAPHTHY	1. 3440945	0.	i,
PLUOPENE	7. 4576271	. 1. 475	11.
ACEHAPTH	8. 5003891	•.	0.
PHENANTH	0. 3664695	29. 216049	19.79679
ANTHRACENE	0. 006445	231.86517	1. 494382
FLUORANTH	2. 4029197	0.5445151	1. 3004261
PYRENE	0. 4881497	9. 1090405	4, 4469697
CHRYSENE	8.437814	15. 493976	6. 7710843
BZ(B)FLUDR	1.0271903	1.9891463	1. 8365854
9Z/K>FLUOR	1. 2619392	9.4859353	0.6132200
9(a)PYPEHE	1.586558	1. 2654639	2.807732
DBZ(AM)ANT	12, 442797	95673877	7. 0588942
I (CD>PYR	8. 9275956	1.5947712	1.4793828
		3	

---REPORT---

CHRONATOGRAM NUMBER 154. LEVEL 18. ***PERKIN ELMER LC-75*** STANGARD WATER UNCLEAMED

COMPOUND	CONG(u4/L)	PESPONSE	RATIO
HHX	28. 297539	7635.	14. 739382
• DX	48. 789814	3859.	7. 44 98869
THE	38. 291212	7723.	14. 909266
DNE	37. 413637	624A.	12. 646332
TETRYL	0.	•.	0.
DHP	56. 787893	3649.	7.0444015
THT	28, 694355	2112.	4. 8772281
2, 5 BHT	37, 422224	2913.	3. 9861884
2,4 BMT	31.954576	2318.	4. 4749933
ONT	A,	518.	1.
NAPHT	58. 988199	3307.	5, 3841699
ACEHAPHTHY	112.429A	7414.	6.5907336
FLUORENE	2. 0241496	440.	8, 8494298
	37.753739	1296.	2, 4826255
ACEHAPTH		3469.	6.6969112
PHEHANTH	24, 254348	133.	9. 2567568
ANTHRACENE	1 .		3. 1776062
FLUORANTH	7. 55K93A2	1846.	2, 2664093
PASENE	32.935163	1174.	1. 0049421
CHRABENE	1,9729443	562.	1, 3127413
92/8/FLU09	2.0659135	680.	
BZCK)FLUOR	4. 9286783	A72.	1.6033977
B(B) PYREHE	1.664967	779.	1,503861
DBZ(AM)ANT	38. 89955	5073.	11.337838
[(CD)PYR	5.377885	479.	1.2100100

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WHAT LEVEL

THE STATE ON TRESPONSE AT LC-75

STORE ON TRESPONSE AT 25400

PESPONSE RATIOS LEVEL 5.
```

COMPOUND	したーアラノクラ4
XMM	8. 8299453
904	7. 72624AR
THE	4, 477834
DHB	2, 3721791
ETRYL	A.
HP	2. 2671351
THT	
	2.7718938
2, 6 DHT	2.3902681
2,4 DHT	1. 5642592
OHT	1.0921598
HAPHT	2.4989782
ACEHAPHTHY	1.6818546
FLUOPENE	23. 233918
ACEHAPTH	9.6688679
PHENANTH	8.4434122
PHTHRACEHE	9.
FLUGRANTH	2. 9672892
PYPENE	0.5888885
CHRYSENE	0.4952439
92(8) FLU09	1, 209555
9Z(K)FLUOR	1.5893414
B(B)PYREHE	2.1527892
DBZCHH)AHT	15. 573191
I (CD) PYR	1. 1622952

RATIOS WITHOUT ONT

COMPOUND	LC-75/254	254/FL	LC-75/FL
HRK .	F, 57F24B3	θ.	●.
RDX	3. A51 <i>6627</i>	A	0.
THE	3.6871918	0.	9.
DHS	1, 9427255	A.	0.
TETRYL	9.	●.	•.
DHP	1,8567954	9.	ē.
THT	2.2788855	9.	0.
2,6 BHT	1.9575472	.	Ō.
2,4 DHT	1, 2810743	9.	ě.
CHT	9. 8287343	9.	Ď.
HAPHT	2.0456333	21.818182	44. 636364
ACENAPHTHY	1.377381	9.	0.
FLUORENE	19.827778	0.	0.
-ACEHAPTH	9. 5471246	78. 25	42. 8125
PHEHANTH	9. 3631393	31. 5	11. 438889
ANTHRACENE	0.	191.6	0.
FLUORANTH	2.4381875	8. 5622733	1.3663845
PYREHE	A. 4816176	9. 16	3, 93
CHRYSENE	9.4055877	16. 21875	6. 578125
BZ(B)FLUOR	A. 9985838	1.0641283	1.0541882
BZCK>FLUOR	1. 3816158	A. 4261668	0.5547854
B(B)PYREHE	1.7629938	1.2895442	2, 2734584
DEZCHHIRNT	12.753996	9. 623629 <i>?</i>	7. 953715
T CODYPYR	0.9518797	1.6181693	1.5326877

```
LIST
   10 REM HPLC SCREEN BATA REBUCTION
      !!!
!"IF LC-75 THPUT 1"
      !"IF 254 nm THPUT 2"
   50 1"IF FLUORESCENCE THPHT 3"
      INPUT FI
   50
   65 ! " WHAT LEVEL IS THE SAMPLE"
   66 INPUT J
   78 !"HON MANY COMPOUNDS"
   80 INPUT A
   90 !"THE CHROMATOGRAM NUMBER IS";
  100 INPUT T
  195 ! "ENTER 1 FOR NATURAL, 2 FOR STANDARD";
  107 INPUT B
  198 ! "ENTER 1 FOR UNCLEANED. 2 FOR CLEANED";
  189 INPUT B
  118 ! THE RESPONSE OF ONT IS";
115 INPUT X
  129 ! "ENTER PEAK HEIGHTS"
  122 IF FI=2 THEN GOTO 1899
  123 IF FI=3 THEN GOTO 1969
  138 FOR I=1 TO R
  148 !$5.03H1([);H2([);;
  141 INPUT B1(I)
  150 HEXT
  160 FOR I=1 TO A
  152 C(I) = ((91(I)/X) + KB(I) + KC(I)) + B. 2
  165 IF 81(I)=0 THEN C(I)=0
  168 IF C(1) CB THEN C(1)=8
  173 R(I)=81(I)/X
  174
      P([)=81([)
  175 NEXT
  180
      111
  195 | TAB39" *** REPORT ***
  187 !!!
      !TAB23"CHROMATOGRAM NUMBER";T
  190
      TRESS'LEVEL"; J
IF FL=1 THEH TTRESS"+**PERKIN ELHER LC-75***
  195
  200
  218
      IF FI=3 THEN !TAB25" *** PERKIN ELMER FLUORESCENCE ***
  228
      IF 9=1 AND D=1 THEN !TAB24"NATURAL WATER UNCLEANED"
  230
      IF 8=1 AND D=2 THEN !TAB27"HATURAL WATER CLEANED"
IF 8=2 AND D=1 THEN !TAB24"STANDARD WATER UNCLEANED"
  248
  250
      IF 9=2 9ND D=2 THEN 1TAB 27"STANDARD WATER CLEANED"
  268
  278
      11
      ! "COMPOUND"; TAB15"CONC(us/L) "TAB35"RESPONSE "TAB55"RATIO"
  288
  283 FOR I=1 TO A
  285 !#5. 03H1([); H2([);
      TRBSC([)TRB25P([)TRB44R([)
  286
  287 NEXT
  290 1111
  380 END
  350
      END
  400 FOR I=1 TO 24
  418 ##5. 83H1([); H2([)
  428 HEXT
  430 END
  440 I"WHAT FILE"
  442 IMPUT FI
      ! PENTER H. Y. DETECTION LIMITS .
  445
```

```
446 FOR (=1 TO 24
 447 1$5.83H1(I);H2(I);;
 448 INPUT
           D(I)
 449 NEXT
 450 FOR T=1 TO 24
451 IF KB(T)=8 THEN KB(T)=1
 452 X([)=D([)*5
 453 A([)=(X([)-KC([))/KB([)
 454 IF X(I)=0 THEN A(I)=0
 455 IF FI=2 THEN S([)=A([)+580
 456 IF FI=1 THEN S(I)=A(I)+410
 457 IF FI=3 THEN S([)=A([)
 458 IF X(I)=0 THEN A(I)=0
 460 NEXT
 465 !!!
 470 !TRB35****REPORT***
 475 !!!
 490 ! "ANALYTE TAB20 INTEGRATOR COUNTS TAB50 NANOGRAMS"
 485 FOR I=1 TO 24
 490 !#5.03H1([);H2([);
 500 !TR813S([)TR842X([)
 510 NEXT
 529 END
 500 !"WHAT FILE"
 610 I"[NPUT 1 FOR LC-75 "
 628 !"[NPUT 2 FOR 254mm "
 630 !"[HPUT 3 FOR FLUORESCENCE"
 648 INPUT FI
 659 1"HOW MANY CALIBRATION CURVES"
 660 INPUT A
 678 ! "COMPOUND TAB28 SLOPE TAB48 INTERCEPT TAB55 CORR COEFF"
 673 A=24
 680 FOR T=1 TO A
 698 1$5,83H1(I);H2(I);
 691 !TAR7KB(T)TAB29KC(T)TAB45Gu(T)
 798 NEXT
 785 !!!
 710 END
 992 !!!
995 ! "WHAT FILE"
 997
    INPUT FE
1000 DIM C(50)
1885 DIM R(58)
1006 ! "WHAT COMPOUND HUMBER"
1007 INPUT X
1009 I"LIST CONC. RESPONSE"
1010 FOR I=1 TO 50
1815 H=[
1020 INPUT C(I); R(I)
1030 IF C(I) AND R(I) <0 THEN GOTO 1200
1848 HEXT
1200 H=H-1
1210 S=0: T=0: F=0: D=0
1220 FOR I=1 TO N
1230 S=S+C(I)
1268 T=T+R([)
1290 F=F+C([)++2
1320 D=D+C(I)+R(I)
1338 NEXT
1348 A=D-(S#T/N)
1350 B=F-(8*#2/N)
1360 KR(K)=R/R
                                      . 7
```

1.

```
1379 KC(X)=(S-KB(X)*T)/N
1375 7=9
1380 FOR T=1 TO N
1398 7=7+0(11**2
1400 NEXT
1418 H(X)=KB(X)+SDR(Z/N-(T/N)++2)/SDR(F/N-(S/N)++2)
1,438 Gn(X)=1/H(X)
1435 !"SLOPE"TAB20"[NTERCEPT"TAB45"CORR COEFF"
1440 !KB(X); TRR2RKC(X); TRB45Gu(X)
1445 !!!!
1446 END
1495 ! "WHAT LEVEL"
1497 THPUT R
1499 !!!
1500 !"ENTER ONT RESPONSE AT LC-75"
1501 INPUT X
1502 ! "ENTER ONT RESPONSE AT 254nm"
1503 INPUT Y
1510 !TAB20"RESPONSE RATIOS"TAB45"LEVEL"; R
1526 !TAB25" *** RATIOS WITH ONT ***
1525 !!
1530 !"COMPOUND"TAB17"LC-75/254"
1560 FOR I=1 TO 24
1579 IF 82(I)=0 THEN 82(I)=-9
1589 T([)=(81([)/X)/(82([)/Y)
1690 V(I)=81(I)/82(I)
1720 IF 83(I)=0 THEN 83(I)=-9
1739 H(I)=82(I)/83(I)
1770 S(I)=81(I)/83(I)
1798 NEXT
1781 FOR I=1 TO 24
1782 IF 82(I)(8 THEN T(I)=8
1783 [#5.03H1(I);H2(I);
1794 !TABZT([)
1785 NEXT
1790 !!!
1800 !TAB22" ** * RATIOS WITHOUT ONT ** **
1810 !!
1820 !"COMPOUND"TAB17"LC-75/254"TAB34"254/FL"TAB51"LC-75/FL"
1838 FOR [=1 TO 24
1835 IF 82(1)(0 THEN H(1)=0
1836 IF B2([)(0 THEN V([)=0
1837 IF 83([)(8 THEN H([)=0
1838 IF 83(I)(0 THEN $(I)=0
1940 !#5.93H1([);H2([);
1841 !TAB7V([)TAB24H([)TAB41S([)
1850 NEXT
1868 END
1899 FOR I=1 TO A
1900 !#5.03H1([);H2([);;
1901 INPUT 82(I)
1902 NEXT
1993 FOR I=1 TO A
1984 C(I)=((82(I)/K)+KB(I)+KC(I))+8.2
1907 IF 82(1)=0 THEN C(1)=0
1911 IF C(1)C0 THEN C(1)=0
1917 R([)=82([)/X
1928 HEXT
1927 !!!
1928 FOP I=1 TO A
1929 P(I)=82(I)
```

```
1930 NEXT
1932 GOTO 185
1969 FOR (=1 TO A
1970 !$5.03H1([);H2([);;
1971 INPUT 83(I)
1972 NEXT
1973 X=1
1974 FOR I=1 TO A
1975 C(I)=((B3(I)/X)*KB(I)+KC(I))*0.2
1980 IF B3(I)=0 THEN C(I)=0
1983 IF C([)(0 THEN C([)=0
1992 R(I)=83(I)
1996 P(I)=83(I)
1997 NEXT
1998 GOTO 185
2082 GOTO 992
2086 GOTO 15
2090 GOTO 1495
```

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APPENDIX D
ANALYTICAL METHODS

APPENDIX D

TABLE OF CONTENTS

PETN, HMX, AND RDX IN WATER SAMPLES

PETN, HMX, AND RDX IN SOIL SAMPLES

HMX IN WATER SAMPLES

HMX IN SOIL SAMPLES

DPA IN SOIL AND SEDIMENT SAMPLES

UDMH IN WATER SAMPLES

ATNBA IN WATER SAMPLES

ATNBA IN SOIL SAMPLES

35DNP IN WATER SAMPLES

35DNP IN SOIL SAMPLES

35DNA IN WATER SAMPLES

35DNA IN SOIL SAMPLES

TDGCL IN WATER SAMPLES

TDGCL IN SOIL SAMPLES

HPLC SCREEN OF WATER SAMPLES FOR NITROSUBSTITUTED MUNITION COMPOUNDS AND PAHS

PETN, HMX, AND RDX IN WATER SAMPLES

PETN, HMX, AND RDX IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for PETN, HMX, and RDX.

A. TESTED CONCENTRATION RANGE

The tested concentration ranges in natural and standard water are listed below:

Analyte	Range (ug/L)
PETN	1.58 to 31.6
HMX	0.43 to 8.5
RDX	1.26 co 25.2

B. SENSITIVITY

The normalized responses (integrator counts) at the natural water detection limits designated in Section 1(C) are listed below:

Analyte	Integrator Counts	Nanograms
PETN	37700	281.1
HMX	121000	143.7
RDX	173000	256.2

The normalized responses (integrator counts) at the standard water detection limits designated in Section 1(C) are listed below:

Analyte	Integrator Counts	Nanograms
PETN	27179	213.1
HMX	96096	110.4
RDX	68495	91.1

C. DETECTION LIMIT

The detection limits in natural water, calculated according to Hubaux and Vos (1970), are listed below:

Analyte	Detection Limit (ug/L)
PETN	4.5
HXX	2.3
RDX	4.1

The detection limits in standard water, calculated according to Hubaux and Vos (1970), are listed below:

Analyte	Detection Limit (ug/L)
PETN	3.4
HMX	1.8
RDX	1.5

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb light at 215 nm and are extractable from water with methylene chloride.

E. ANALYSIS RATE

After instrument calibration, one snalyst can analyze 10 extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
PETN	Pentaerythrite tetranitrate	78-11-5
	Pentaerythritol tetranitrate	
	2,2-Bis[(nitrooxy)-methyl]-	
	1,3-Propenediol dinitrate (ester)	
	Nitropentaerythritol	
	Pentrit	
HMX	Cyclotetramethylenetetranitramine	2691-41-0
	Octahydro-1,3,5,7-tetrasocine	
	1,3,5,7-Tetranitro-1,3,5,7-	
	tetrazacyclooctane	
	Octogen	
RDX	Cyclotrimethylenetrinitremine	121-84-4
	Hexogen, T-4, Cyclonite, Hexahydro-	
	1,3,4-trinitro-s-triasine	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

Analyte PETN	Formula C5H8N4O12	Melting Point (°C)	Boiling Point 180 at 50 torr	Density (g/ml) 1.77
НМХ	C4 H8 N8 O8	276		1.77-1.96*
RDX	C3H6N6O6	204.1		1.816

* There are four polymorphic forms of RMX with this range of densities.

Chemical Structures

PETN

DCX

RDX

C. CHEMICAL REACTIONS

All of these compounds are highly explosive, and caution should be used in handling. Each compound is subject to alkaline hydrolysis in aqueous solution.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

- 1. Detector: Perkin-Elmer LC-75 variable-wavelength detector $(\lambda = 215 \text{ nm})$
- 2. Column: Zorbax-CN (4.6-mm ID \times 25 cm)

Particle size: 7-8 um

- 3. Flow Rate/Mobile Phase: 1 ml/min/35% H₂O/65% methanol
- 4. Temperature: 22°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Times:

Analyte	Retention Time (Minutes)
RDX	7.8
HMX	11.8
PETN	13.9

C. HARDWARE/GLASSWARE

- 1. 1-liter separatory funnel (Teflon® or glass) (8).
- 2. 500-ml K-D flask (8).
- 3. 15-ml K-D receiver (8).
- 4. 3-ball Snyder column (8).
- 5. 2-ball micro-Snyder column (8).
- 6. 10-ml graduated centrifuge tubes (8).
- 7. Disposable glass pipettes.

D. CHEMICALS

- 1. Nanograde methylene chloride--J.T. Baker Company.
- 2. HPLC-grade acetonitrile--J.T. Baker Company.
- 3. HPLC-grade water--J.T. Baker Company.
- 4. Anhydrous sodium sulfate-reagent grade.
- 5. HPLC-grade methanol.

4. STANDARDS

A. CALIBRATION STANDARDS

Separate calibration stock solutions are prepared for each analyte. A composite working calibration standard is prepared from these solutions.

- 1. The RDX stock calibration standard (6,310 ug/ml) is prepared by weighing 63.1 mg of RDX in a 10-ml volumetric flask, dissolving the RDX in a few ml of acetonitrile, and diluting to the mark with acetonitrile. An intermediate RDX stock calibration standard is prepared by pipetting 1 ml of the RDX stock calibration standard into a 100-ml volumetric flask and diluting to the mark with methanol to give a solution containing 63.1 ug/ml of RDX.
- 2. The HMX stock calibration standard (5,320 ug/ml) is prepared by weighing 53.2 mg of HMX in a 10-ml volumetric flask, dissolving the HMX in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and diluting to the mark with acetonitrile. An intermediate HMX stock calibration standard is prepared by pipetting 1 ml of the HMX stock calibration standard into a 50-ml volumetric flask and diluting to the mark with methanol to give a solution containing 106.4 ug/ml of HMX.
- 3. The PETN stock calibration standard (3,950 ug/ml) is prepared by weighing 39.5 mg of PETN in a 10-ml volumetric flask, dissolving the PETN in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and

diluting to the mark with acetonitrile. An intermediate PETN stock calibration standard is prepared by pipetting 1 ml of the PETN stock calibration standard into a 50-ml volumetric flask and diluting to the mark with methanol to give a solution containing 79.0 ug/ml of PETN.

4. Prepare a series of composite working calibration standards by making dilutions of the intermediate calibration standards with 50% methanol/50% water as follows:

Working Calibration Standard	Intermediate Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
В	RDX	5	50
	HMX	1	
	PETN	5	
C	RDX	5	100
	HMX	1	
	PETN	5	
D	Standard B	5	25
E	Standard B	5	50
F	Standard B	5	100

Working Calibration	Concentration (ug/ml)		
Standard	RDX	HMX	PETN
В	6.31	2.13	7.90
C	3.15	1.06	3.95
D	1.26	0.426	1.58
E	0.631	0.213	0.790
F	0.315	0.106	0.395

B. CONTROL SPIKES

 The working control spike solutions are prepared in the same manner as the working calibration standards using the same letter designations for the different solutions; therefore, the Working Control Spike Solution B has the same concentration as the Working Calibration Standard B.

- 2. Pipette 2 ml of the corresponding working control spike solutions into 500 ml of standard or natural water. The solutions used are selected to provide a concentration range of 0.5 to 10 times the desired detection limit.
- 3. Determine the precision, accuracy, and detection limits for each analyte.

Working Control Spike Used	Analyte Concentration in the Working Control Spike Solution (ug/ml)		Spiked Analyte Concentration in Water (ug/L)	
			0.0	
В	RDX	6.31	25.2	
	HMX	2.13	8.5	
С	PETN	7.90	31.6	
	RDX	3.15	12.6	
•	HMX	1.06	4.26	
	PETN	3.95	15.8	
D	RDX	1.26	5.04	
	HMX	0.426	1.70	
E	PETN	1.58	6.32	
	RDX	0.631	2.52	
	HMX	0.213	0.851	
	PETN	0.790	3.16	
F	rdx	0.315	1.26	
	HMX	0.106	0.426	
	Petn	0.395	1.58	

5. PROCEDURE

A. EXTRACTION

- 1. Measure 500 ml of the water sample into a 1-L separatory funnel.
- Check the pH of the sample with pH paper, and adjust the pH to neutral, if necessary.

- 3. Extract the sample sequentially with three 100-ml portions of methylene chloride. After each portion has been added, shake the funnel vigorously for at least 5 minutes.
- 4. Let the layers separate for about 2 minutes after each extraction.
- 5. Draw off the methylene chloride and pass through a glass funnel filled with a small plug of glass wool and about l inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 10-ml K-D receiver.
- 6. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
- 7. Add a boiling chip (Hengar) to the methylene chloride extract in the flask and attach a 3-ball Snyder column to the apparatus.
- 8. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
- 9. The balls of the Snyder column should actively chatter when the solvent is evaporating.
- 10. When the apparent volume of the solution remaining in the receiver is about 1 ml, remove the apparatus from the water bath and allow to cool. After about 1 ml of methylene chloride has drained into the receiver, remove the receiver from the K-D flask.
- 11. Add approximately 2 ml of HPLC methanol to the receiver.

 Attach a 2-ball micro-Snyder column and reconcentrate. When
 the apparent volume in the receiver reaches 0.5 ml, remove
 the receiver from the water bath.
- 12. Repeat Step 11 two times.
- 13. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube rinsing quantitatively with HPLC acetonitrile. Raise the extract

volume to 1.0 ml in the centrifuge tube with HPLC methanol. Dilute to 2 ml with HPLC water.

- 14. Transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
- 15. The extract is now ready for chromatography by HPLC.

B. CALIBRATION

- 1. Inject Working Calibration Standards G, F, E, D, C, and B and a blank singly at the beginning of the analytical run. Inject Working Calibration Standard D at the conclusion of the analytical run to verify constant instrument response.
- 2. Plot the normalized integrator areas versus nanograms/microliter of each standard to obtain a working curve.

C. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample according to the conditions given in Section 3(B).
- 3. Measure the response of the sample for the components of interest.

6. CALCULATIONS

Determine the concentration of RDX according to the following formula:

Concentration (ug/L) =
$$\frac{(A)(V_r)}{V_g}$$

where: A = Concentration (ug/ml) of analyte found in the sample
by comparison with the appropriate standard curve
(ug/ml),

V_r = Volume of total extract (ml), and

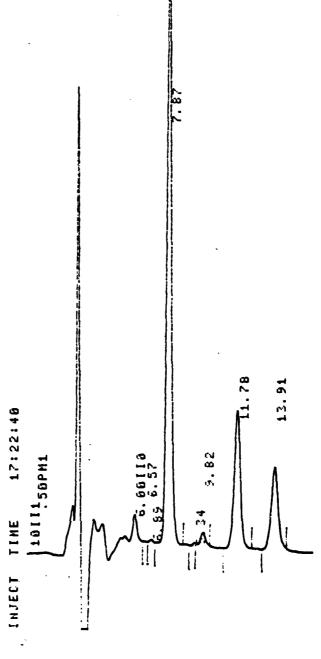
V_a = Volume of initial sample extracted (L).

7. REFERENCES

None found.

8. DATA

See attached data sheets.



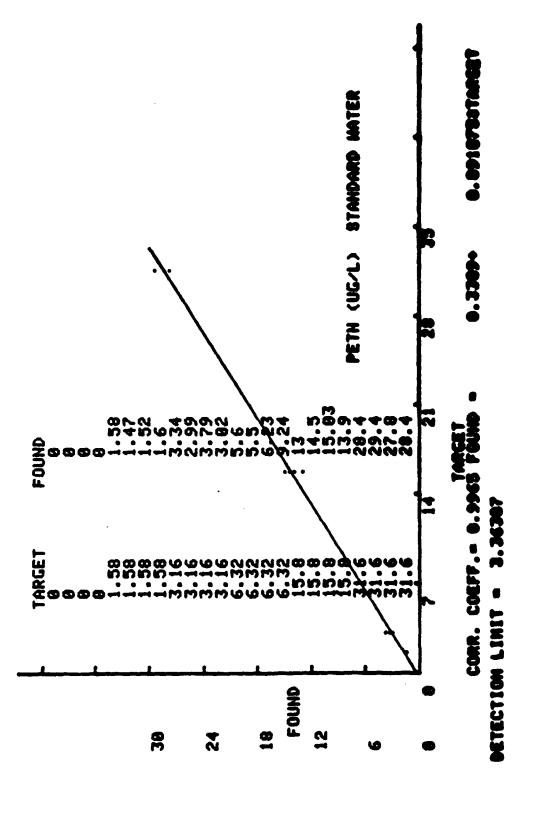
Chromatogram of Standard Water Spiking Experiment

Analyte	Amount Spiked	Retention Time
RDX	12.6 ug/L	7.87 min
HMX	4.3 ug/L	11.78 min
PETN	15.8 ug/L	13.91 min

PETN (UG/L) STANDARD WATER

TARGET CONCENTRATION	1	5 C v A	3	4	
0.0000	0.6000	0.0660	0.0000	C • C C C C	
1.58	1.58	1.47	1.52	1.60	
3.16	3.34	2.99	3.79	3.02	
6.32	5.60	5.50	6 • 23	9.24	
15.ĉ	13.6	14.5	15.0	13.9	
31.6	28.4	29.4	27.8	28.4	

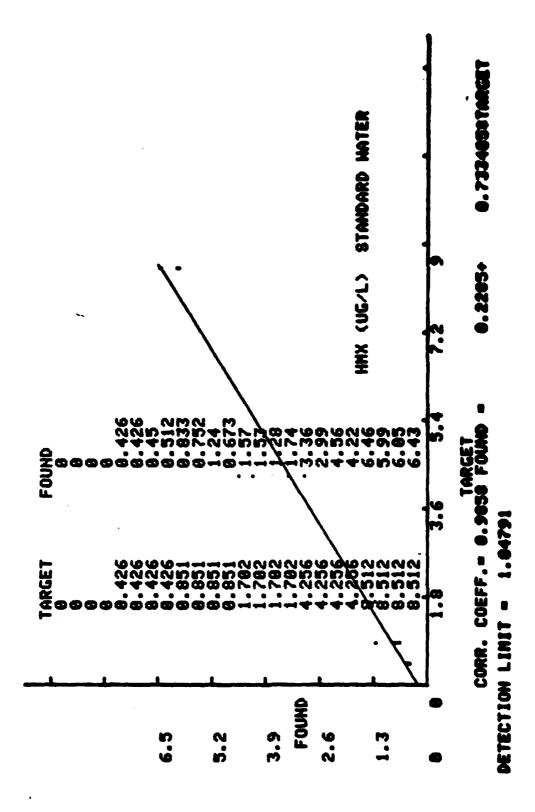
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
(•6006	6 • 6 0 6 5	0.6000	0.6600	0.0606	
1.58	1.54	0.0591	3.83	-2.3734	
3.16	3.28	0.372	11.3	3.96	
6.32	6.64	1.76	26.5	5.10	
15.8	14.1	0.871	6.17	-10.7120	
31.6	28.5	0.663	2.33	-9.6101	



HMX (UG/L) STAMBARD WATER

TAMGET COMCENTRATION	1	DAY 2	3	4	• • • • • • • • • •
0.0000	0.0000	6.0060	6.6000	0.0000	
3.426	0.426	0-426	0.450	0.512	
0.851	0.833	0.752	1.24	0.673	
1.70	1.57	1.53	1.28	1.74	
4 • 2 6	3.36	2.59	4.56	4.22	
8.51	6.46	5.99	6.05	6.43	

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT Inaccuracy	
n.ceae	G • O C O O	0.000	0.0000	0500.0	
0 • 426	0.453	0.0406	8.95	6.46	
0.851	€ •874	0.252	28.5	2.76	
1.70	1.53	0.190	12.4	-10.1058	
4.26	3.78	0.731	19.3	-11.1255	
۴.51	6.23	0.247	3.96	-26.7756	

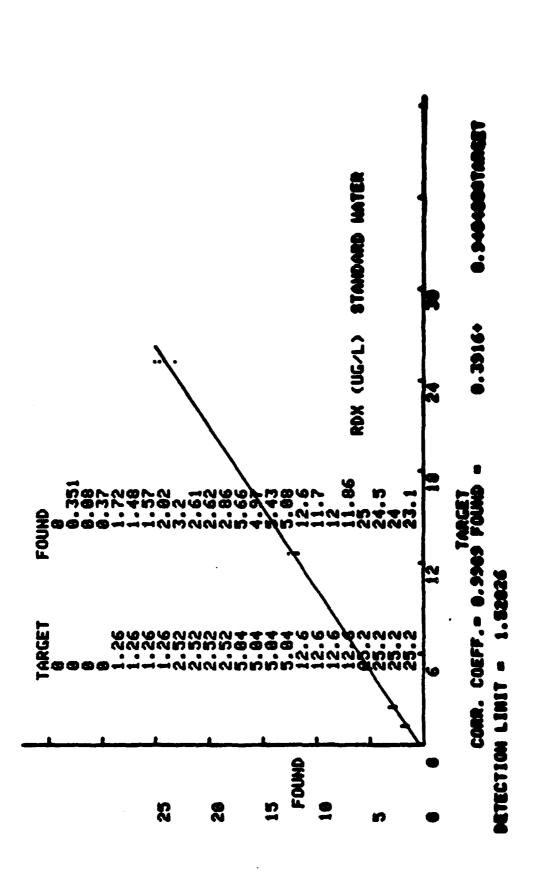


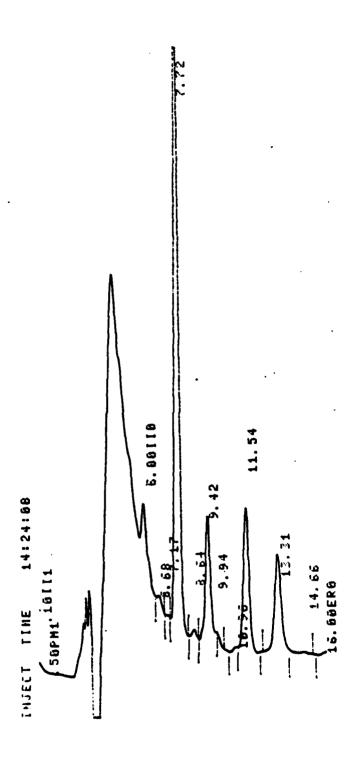
!

RDX (UGZL) STANDARD WATER

TARGET CONCENTRATION	1	DAY 2	3	4	•••••
0 • 0 0 0 0	0.0000	0.351	0.0800	0.370	
1.26	1.72	1.48	1.57	2.02	
2.52	3.20	2.61	2.62	2.86	
5 • 0 4	5.66	4.97	5.43	5.08	
12.6	12.6	11.7	12.0	11.9	
25.2	25.0	24.5	24.0	23.1	

TARGET. CONCENTRATION	AVERAGE FCUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
J.6000	u • 2 0 0	0.188	93.9	0.0000
1.26	1.70	0.237	13.9	34.7
2.52	2.82	0.277	9.81	12.0
5.04	5 • 28	0.318	6.01	4.86
12.6	12.0	0.393	3.26	-4.444
25.2	24.1	0.810	3.36	-4.1667





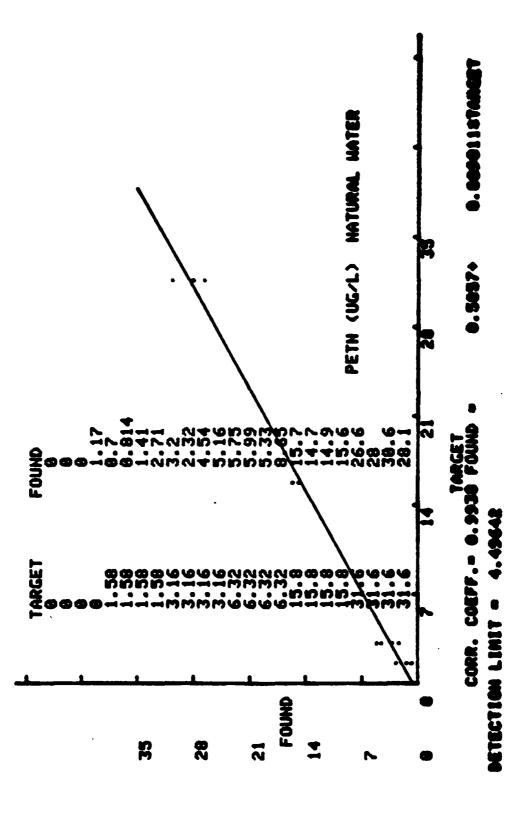
Chromatogram of Natural Water Spiking Experiment

Analyte	Amount Spiked	Retention Time
RDX	12.6 ug/L	7.72 min
HMX	4.3 ug/L	11.54 min
PETN	15.8 ug/L	13.31 min

PETR (UG/L) NATURAL WATER

TARGET CONCENTRATION	1	DAY 2	3	4	
3.0633	0.0006	0.0050	0.0000	1.17	
1.58	0.700	0.814	1.41	2.71	
3.16	3.20	2.32	4.54	5.16	
6.32	5.75	5.99	5.33	8.65	
15.8	15.7	14.7	14.9	15.6	
31.6	26.6	28.0	30.6	28.1	

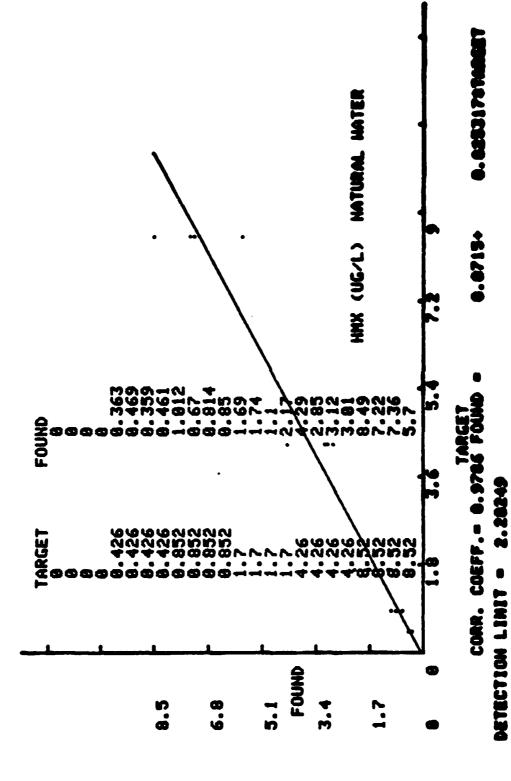
TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	, 9 •2 92	0.585	200	0.0000	
1,58	1.41	0.922	65.4	-10.8544	
3.16	3.80	1.28	33.8	20.4	
• 6.32	6.43	1.50	23.4	1.74	
15.8	15.2	0.499	3.28	-3.6392	
31.6	28.3	1.66	5.87	-10.3639	



HMX (UG/L) NATURAL WATER

TARGET CONCENTRATION	<u>i</u>	DAY 2	3	4	
0.7863	0.0000	0.0000	J.9000	0.0000	
0.426	0.363	0.469	0.359	0.461	
0.852	1.01	0-670	0.814	0.450	
1.79	1.69	1.74	1.10	2.17	
4.26	4.29	2.85	3.12	3.01	
8.52	8.49	7.22	7.36	5.70	

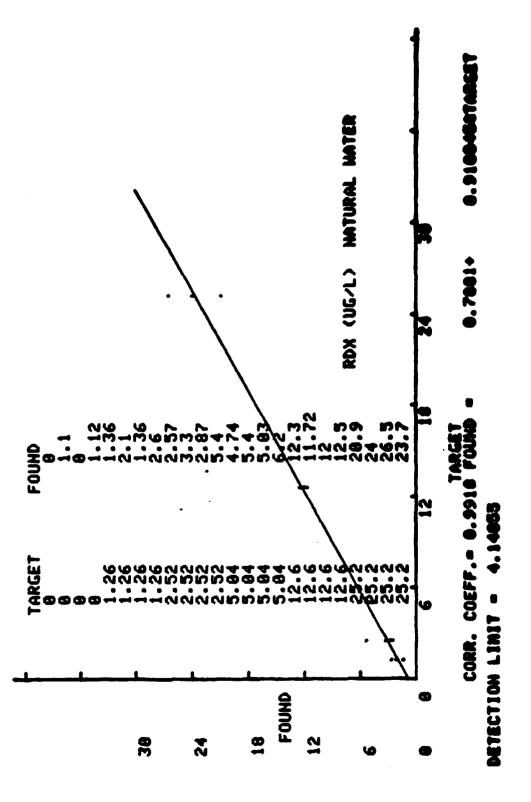
TARGET CONCENTRATION	ÄVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.6300	0.0000	0.000	0.000	0.0000
0.426	0.413	0.0602	14.6	-3.0517
0.852	0.836	0.140	16.8	-1.8193
1.70	1.67	0.440	26.3	-1.4706
4.26	3.32	0.658	19•8	-22.1244
8.52	7.19	1.15	15.9	-15.5810



POX (UC/L) NATUPAL WATER

TARGET CONCENTRATION	1	DAY 2	3	4	
0.000 €	0.0000	1.10	0.0000	1.12	
1.26	1.36	2.10	1.36	2.60	
2.52	2.57	3.30	2.87	5 • 4 G	
5.04	4.74	5.40	5.03	6.20	
12.6	12.3	11.7	12.0	12.5	
25.2	20.9	24.0	26.5	23.7	

TARGET CONCENTRATION	AVERAGE FOUNC VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0 •555	0.641	115	C.0008	
1.26	1.85	0.607	32.7	47.2	
2.52	3.53	1.28	36.2	40.3	
5 • 0 4	5.34	0.632	11.8	6.00	
12.6	12.1	0.342	2.62	-3.7302	
25.2	23.8	2.25	9.64	-5.6548	



PETN, HMX, AND RDX IN SOIL SAMPLES

PETN, HMX, AND RDX IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for PETN, HMX, and RDX.

A. TESTED CONCENTRATION RANGE

The tested concentration ranges in natural and standard soil are listed below:

Analyte	Range (ug/g)
PETN	0.8 to 16.0
HMX	0.79 to 15.8
RDX	0.96 to 19.2

B. SENSITIVITY

The normalized responses (integrator counts) at the natural soil detection limits designated in Section 1(C) are listed below:

Analyte	Integrator Counts	Nanograms
PETN	53,900	191
HMX	354,000	325
RDX	203,000	224

The normalized responses (integrator counts) at the standard soil detection limits designated in Section 1(C) are listed below:

Analyte	Integrator Counts	Nanograms
PETN	54,600	188
HMX	286,000	261
RDX	128,000	147

C. DETECTION LIMIT

The detection limits in natural soil, calculated according to Hubaux and Vos (1970), are listed below:

Analyte	Detection Limit (ug/g)
PETN	2.3
HMX	4.1
RDX	2.7

The detection limits in standard soil, calculated according to Hubaux and Vos (1970), are listed below:

Analyte	Detection Limit (ug/g)
PETN	2.4
HMX	4.6
RDX	1.9

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb light at 215 nm and are extractable from soil with methylene chloride/acetone. Interferences are minimized by silica-gel cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyse 10 extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS)
REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
PETN	Pentaerythrite tetranitrate	78 - 11 <i>-</i> 5
	Pentaerythritol tetranitrate	
	2,2-Bis[(nitrooxy)-methy1]-	
	1,3-Propenediol dinitrate (ester)	

Analyte	Alternate Nomenclature	CAS Registry Number
	Nitropentaerythritol	
	Pentrit	
нмх	Cyclotetramethylenetetranitramine	2691-41-0
	Octahydro-1, 3, 5, 7-tetrazocine	
	1,3,5,7-Tetranitro-1,3,5,7-	
	tetrazacyclooctane	
	Octogen	
RDX	Cyclotrimethylenetrinitramine	121-84-4
	Hexogen, T-4, Cyclonite, Hexahydro-	
	1,3,4-trinitro-s-triazine	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

Analyte PETN	Formula C5H8O12N4	Melting Point (°C) 141	Point 180 at 50 torr	Density (g/ml) 1.77
нмх	C4H8O8N8	276		1.77-1.96*
RDX	C3H6O6N6	204.1		1.816

* There are four polymorphic forms of HMX with this range of densities.

Chemical Structures

PETN

HMX

RDX

C. CHEMICAL REACTIONS

All of these compounds are highly explosive, and caution should be used in handling. Each compound is subject to alkaline hydrolysis in aqueous solution.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

- 1. Detector: Perkin Elmer LC-75 variable-wavelength detector ($\lambda = 215 \text{ nm}$)
- 2. Column: Zorbax-CN (4.6-mm ID x 25 cm)
 Particle size: 7-8 um

3. Flow Rate/Mobile Phase: 1 ml/min

35% H₂O/65% methanol

4. Temperature: 22°C

5. Injection Volume: 20 ul, fixed loop

6. Retention Times:

Analyte	Retention Time (Minutes)
RDX	5.4
HMX	6.7
PETN	7.7

C. HARDWARE/GLASSWARE

- 1. 50-liter centrifuge tubes with Teflon -lined screw caps (8);
- 2. 500 ml K-D evaporative flasks (8);
- 3. 10-ml graduated K-D receivers (8);
- 4. 3-ball Snyder column (8);
- 5. 2-ball micro-Snyder column (8);
- 6. 15-ml graduated centrifuge tubes (8); and
- 7. 10-ml glass or polyethylene syringes with Luer-lock attachments (10).

D. CHEMICALS

- 1. Nanograde methylene chloride;
- 2. HPLC-grade methyl alcohol--J.T. Baker Company;
- 3. HPLC-grade water--J.T. Baker Company;
- 4. Anhydrous sodium sulfate--reagent grade;
- 5. Nanograde hexane;
- 6. Nanograde acetone; and
- 7. Silica-Gel Sep-Paks -- Waters Associates.

4. STANDARDS

A. CALIBRATION STANDARDS

Separate calibration stock solutions are prepared for each analyte. A composite working calibration standard is prepared from these solutions.

- The RDX stock calibration standard (1.92 mg/ml) is prepared by weighing 47.9 mg of RDX into a 25-ml volumetric flask, dissolving the RDX in a few ml of acetonitrile, and diluting to the mark with acetonitrile.
- 2. The HMX stock calibration standard (7.91 mg/ml) is prepared by weighing 79.1 mg of HMX in a 10-ml volumetric flask, dissolving the HMX in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and diluting to the mark with acetonitrile. An intermediate HMX stock calibration standard is prepared by pipetting 5 ml of the HMX stock calibration standard into a 50-ml volumetric flask and diluting to the mark with acetonitrile to give a solution containing 791 ug/ml of HMX.
- 3. The PETN stock calibration standard (8.0 mg/ml) is prepared by placing the entire SARM solution (200 mg PETN) in a 25-ml volumetric flask and diluting to the mark with acetonitrile. An intermediate PETN stock calibration standard is prepared by pipetting 5 ml of the PETN stock calibration standard into a 50-ml volumetric flask and diluting to the mark with acetonitrile to give a solution containing 800 ug/ml of PETN.
- 4. Prepare a series of composite working calibration standards by making dilutions of the intermediate calibration standards for PETN and HMX and the stock calibration standard for RDX. Dilute with 50% methanol/50% water as follows:

		Volume	
Working Calibration Standard	Standard Diluted	of Standard Used (ml)	Final Volume (ml)
В	RDX (stock)	1	25
	HMX	2	
	PETN	2	
С	RDX (stock)	1	50
	HMX	2	
	PETN	2	
D	Standard B	5	25
E	Standard B	5	50
F	Standard B	5	100

Working Calibration Standard	Concentration (ug/ml)			
	RDX	HMX	PETN	
В	76.8	63.3	64	
С	38.4	31.6	32	
D	15.3	12.7	12.8	
E	7.7	6.3	6.4	
F	3.8	3.2	3.2	

B. CONTROL SPIKES

- 1. Prepare Control Spike Solution A for RDX by diluting 5 ml of the calibration standard stock (concentration 1.92 mg/ml) to 50 ml with acetone.
- 2. Prepare Control Spike Solution B for RDX by diluting 5 ml of the stock control spike solution to 50 ml with acetone.
- 3. Prepare Control Spike Solution C for HMX and PETN by combining 1 ml each of the calibration standard stock for HMX and the calibration standard stock for PETN in a 25-ml volumetric flask and diluting to volume with acetone.
- 4. Prepare Control Spike Solution D for HMX and PETN by diluting 5 ml of Control Spike Solution C to 50 ml with acetone.

Control Spike Solution	Concentration (ug/ml)
A (RDX)	192
B (RDX)	19.2
C (HMX, PETN)	316, 320
D (HMX, PETN)	31.6. 32

Control Spike Solution	Standard Diluted	Dilution (ml)	Final Volume (ml)	
A (RDX)	Stock Calibration	5	50	
B (RDX)	Control Spike A	5	50	
C (HMX, PETN)	Stock Calibration	1, 1	25	
D (HMX, PETN)	Control Spike C	5	50	

- 5. Allow the soil sample to air dry on the dull side of aluminum foil until it can be sieved through a 30-mesh sieve. (Sediment samples are extracted wet.)
- 6. Weigh 20 g of sieved soil or wet sediment into a 50-ml centrifuge tube with a Teflon®-lined screw cap.
- 7. Pipette a known amount of the control spike solutions for RDX, HMX, and PETN onto the 20-g soil sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

RDX Control	Spike Volume	Concentration of Spiked Soil (ug/g)
Spike Solution	(m1)	RDX
В	1.0	0.96
В	2.0	1.9
В	4.0	3.8
A	1.0	9.6
A	2.0	19

HMX/PETN Control Spike	Spike Volume	Concentration of Spiked Soil (ug/g)		
Solution	(ml)	HMX	PETN	
D	0.5	0.79	0.80	
D	1.0	1.6	1.6	
D	2.0	3.2	3.2	
C	0.5	7.9	8.0	
С	1.0	16	16	

8. Allow the soil to air dry for at least 1 hour. Shake the soil to ensure mixing of the spiking solution throughout the sample.

5. PROCEDURE

A. SAMPLE PREPARATION

- 1. Let all soil samples air dry on the dull side of aluminum foil until they are suitably dry so they can be sieved through a 30-mesh sieve.
- 2. Sample the sieved soil by quartering and weighing 20 g into a 50-ml centrifuge tube.

B. EXTRACTION

- 1. Add 35 ml of 20% acetone in methylene chloride to the centrifuge tube.
- 2. Cap the tube and shake for 3 to 5 minutes.
- 3. Extract the sample sequentially with three 35-ml portions of the methylene chloride/acetone mixture.
- 4. Decant off the methylene chloride/acetone mixture each time and pass through a glass funnel filled with a small plug of glass wool and approximately 1 inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 10-ml K-D receiver.
- 5. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.

- 6. Add a boiling chip (Teflon®) to the methylene chloride extract in the flask, and attach a 3-ball Snyder column to the apparatus.
- 7. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
- 8. The balls of the Snyder column should actively chatter when the solvent is evaporating.
- 9. When the apparent volume of the solution remaining in the receiver is about 1 ml, remove the apparatus from the water bath and allow to cool. After about 1 ml of solvent has drained back into the receiver, remove the receiver from the K-D flask.
- 10. Add approximately 2 ml of nanograde hexane to the receiver.

 Attach a 2-ball micro-Snyder column and reconcentrate. When
 the apparent volume in the receiver reaches 0.5 ml, remove
 the receiver from the water bath.
- 11. Repeat Step 10 twice.
- 12. Detach the micro-Snyder column from the receiver. With a dispo pipette, transfer the extract into a 10-ml glass syringe fitted with a silica-gel Sep-Pak*. Rinse the receiver three times with 2 ml of 20% methylene chloride in hexane solution, transferring each rinse to the 10-ml syringe. Set aside the receiver for later use.
- 13. Pass the combined rinses through the silica-gel Sep-Pak® at a rate of approximately 1 to 2 ml/min, discarding the eluate.
- 14. Quantitatively rinse the K-D receiver from Step 12 three times with a total of 1 to 2 ml of 50% methanol in methylene chloride solution, transferring each rinse to the 10-ml syringe fitted with the silica-gel Sep-Pak[®]. Add 50% methanol in methylene chloride to the syringe to make a total volume of 10 ml.

- 15. Elute the silica-gel Sep-Pak[●], with the 10-ml total volume of 50% methanol in methylene chloride, into another 10-ml K-D receiver at a rate of 1 to 2 ml/min.
- 16. Add a Teflon® boiling chip to eluate, attach a 2-ball micro-Snyder column, and concentrate the sample on a water bath heated to 80°C. When the apparent volume of the solution is about 0.5 ml, remove the apparatus from the water bath.
- 17. Detach the micro-Snyder, and add approximately 2 ml of HPLC methanol to the receiver. Reconcentrate the sample to 0.5 ml.
- 18. Repeat Step 17 twice.
- 19. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube rinsing quantitatively with HPLC methanol. Raise the extract volume to exactly 2.5 ml in the centrifuge tube with HPLC methanol. Dilute to 5 ml with HPLC water.
- 20. Transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
- 21. The extract is now ready for chromatography by HPLC.

C. ANALYSIS

- 1. Inject 20 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample using the conditions given in Section 3(B).
- 3. Measure the response of the component of interest.

6. CALCULATIONS

A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.

AMD.2/MERT4.12 07/20/82

B. Determine the concentration of RDX, HMX, and PETN according to the following formula:

Concentration (ug/g) =
$$\frac{(A)(V_t)}{W_s}$$

 V_t = Volume of total extract (m1), and

W_s = Weight of initial sample extracted (g).

7. REFERENCES

None found.

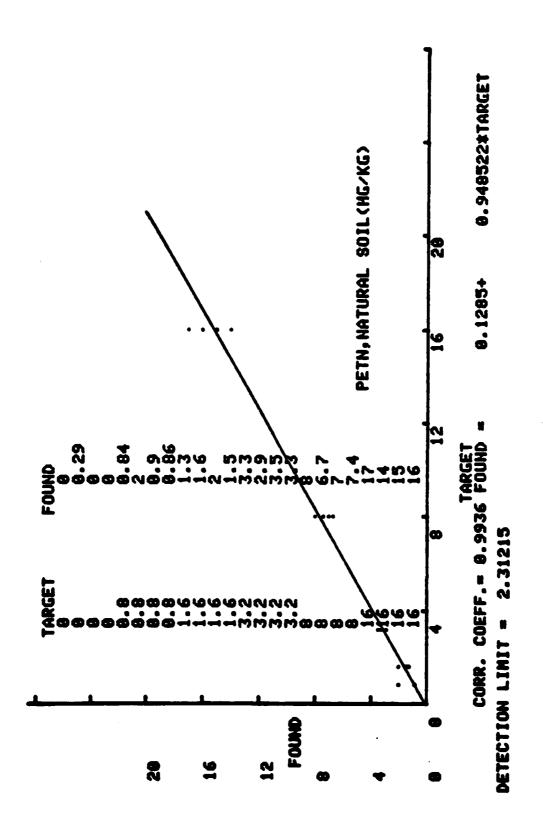
8. DATA

See attached data sheets.

PETN + NATURAL SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.000	0.000	0.290	0.0000	0.0000	
0.800	C • 84 O	2.00	0.900	0.860	
1.60	1.30	1.60	2.00	1.50	
3.20	3.30	2.90	3.50	3.30	
8.00	8.00	6.70	7.00	7.40	
16.0	17.0	14.0	15.0	16.0	

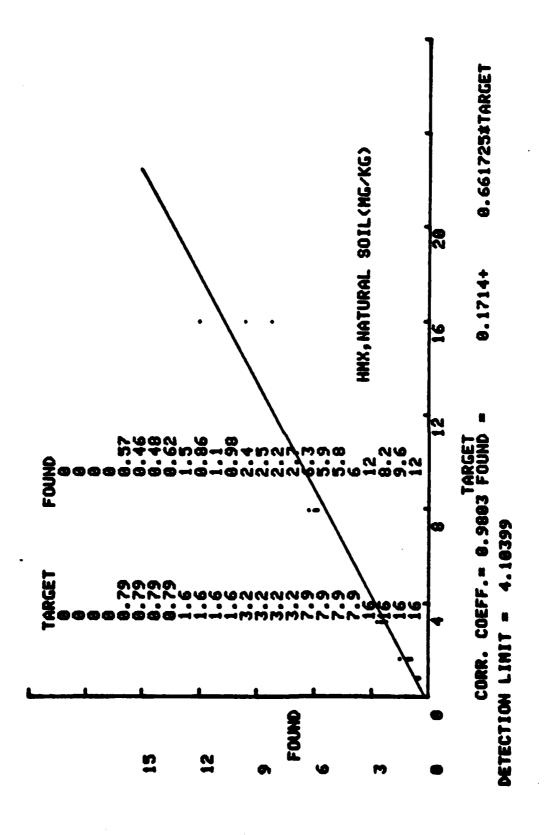
TARGET CONCENTRATION	AVEPAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT Inaccuracy
0.0000	0.0725	0.145	200	0.0000
0.809	1.15	0.567	49.3	43.7
1.60	1.60	0.294	18•4	0.0000
3.20	3.25	0.252	7.74	1.56
8.00	7.27	0.562	7.73	-9.0625
16.0	15.5	1.29	8.33	-3.1250



HMX . NATURAL SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	0.0000	
0.790	0.570	0.460	0.480	0.620	
1.60	1.50	0.860	1.10	0.980	
3.20	2.40	2.50	2.20	2.70	
7.90	6.30	5.90	5.80	6.00	
16.0	12.0	8.20	9.60	12.0	

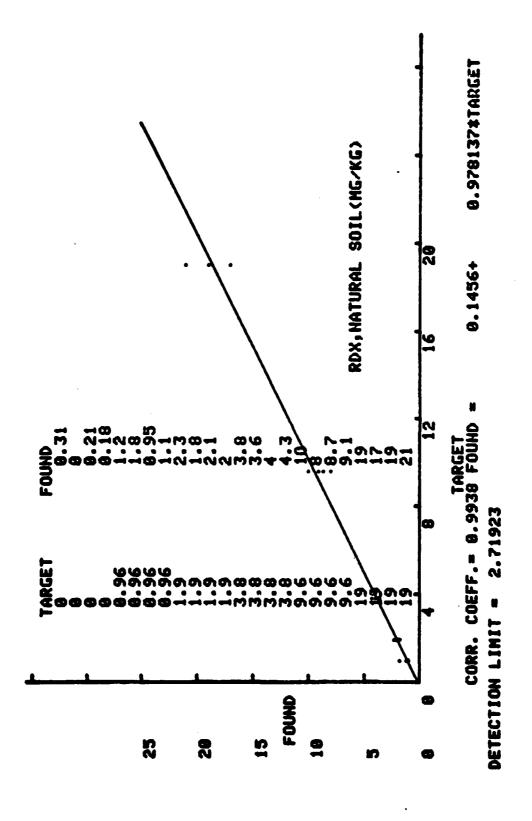
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.000
0.790	0.532	0.0754	14.2	-32.5949
1.60	1.11	0.278	25.0	-30.6250
3.20	2.45	0.208	8.50	-23.4375
7.90	6.00	0.216	3.60	-24.0506
16.0	10 • 4	1.88	18.0	-34.6875



ROX . NATURAL SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.310	0.0000	0.210	0.180	
0.960	1.20	1.80	0.950	1.10	
1.90	2.30	1.80	2.10	2.00	
3.80	3.80	3.60	4.00	4.30	
9.60	10.00	8.00	8.70	9.10	
19.0	19.0	17.0	19.0	21.0	

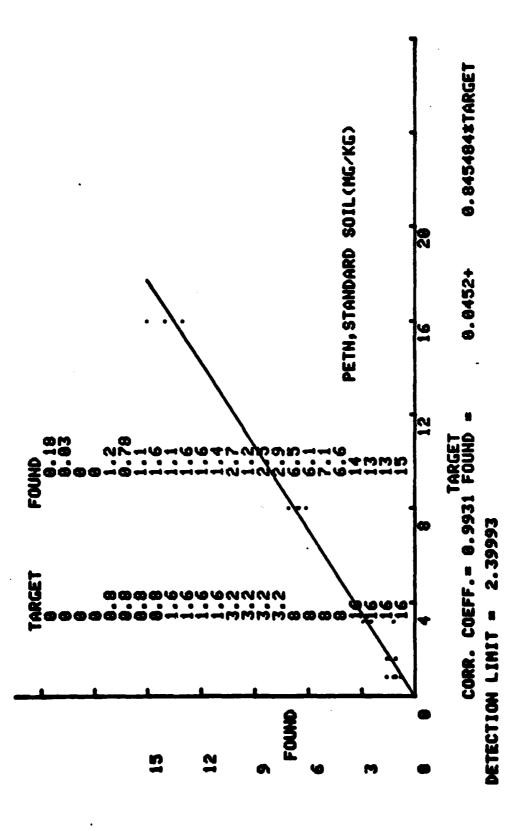
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT Imprecision	PERCENT INACCURACY
0.0000	0.175	0 • 129	73.8	0.0000
0.960	1.26	0.373	29.5	31.5
1.90	2.05	0.208	10.2	7.89
3.80	3.92	0.299	7.61	3.29
9.60	8.95	0.835	9.33	-6.7708
19.0	19.0	1.63	8.59	-0.0000



PETN+STANDARD SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.000	0.180	0.0300	0.0000	0.0000	
0.800	1.20	0.780	1.10	1.60	
1.60	1.10	1.60	1.60	1.40	
3.20	2.70	1.20	2.50	2.90	
8.00	6•5Ó	6.10	7.10	6.60	
16.0	14.0	13.0	13.0	15.0	

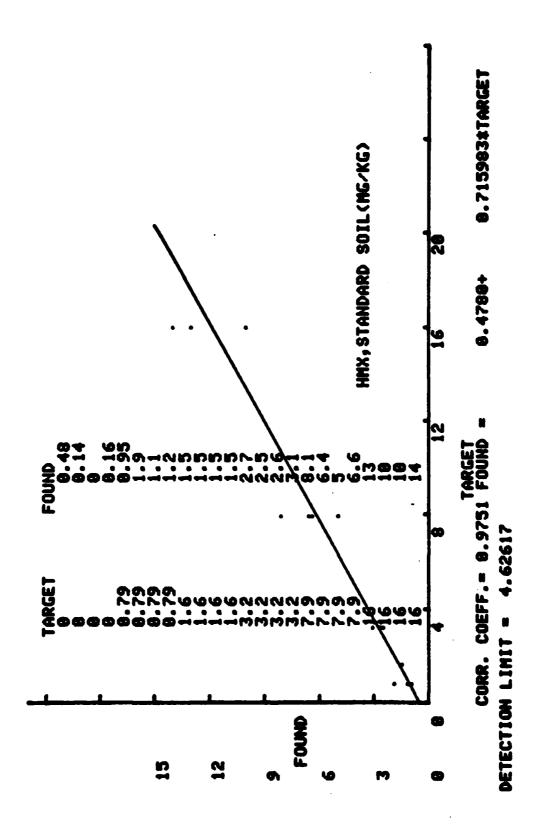
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0009	0.0525	0.0862	164	0.0000
0.800	1.17	0.338	28.9	46.2
1.60	1.42	0.236	16.6	-10.9375
3.20	2.32	0.768	33.0	-27.3438
8 • 0 0	6.57	0.411	6.26	-17.8125
16.0	13.8	0.957	6.96	-14.0625



HMX.STANDARD SOIL(MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.480	0.140	0.0000	0.160	
0.790	0.950	1.90	1.10	1.20	
1.60	1.50	1.50	1.50	1.50	
3.20	2.70	2.50	2.60	3.10	•
7.90	8.10	6 • 40	5.00	6.60	
16.0	13.0	10.00	10.00	14.0	

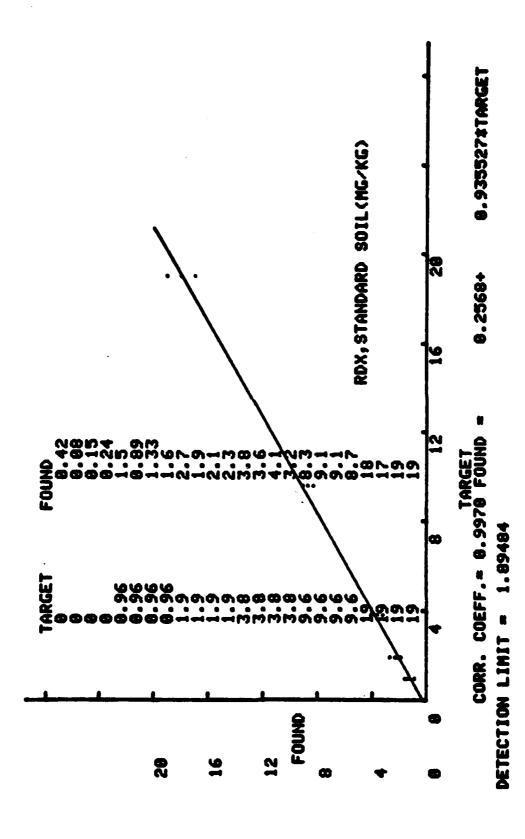
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
C • 0 0 C O	0.195	0.203	104	0.0000
0.790	1.29	0.421	32.7	63.0
1.60	1.50	0.0000	0.0000	-6.2500
3.20	2.72	0.263	9.65	-14.8438
7.90	6.52	1.27	19.4	-17.4051
16.0	11.8	2.06	17.5	-26.5625



RDX.STANDARD SOIL

TARGET CONCENTRATION		1	DAY 2	3	4
0.0000		0.420	0.0800	0.150	0.240
0.960		1.50	0.890	1.33	1.60
1.90		2.70	1.90	2.10	2.30
3.80		3.80	3.60	4.10	3.20
9.60	_	8.30	9.10	9.10	8.70
19.0		18.0	17.0	19.0	19.0

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0.222	0.147	66•1	0.0000	
C.960	1.33	0.314	23.6	38.5	
1.90	2.25	0.342	15.2	18.4	
3.80	3.67	0.377	10.3	-3.2895	
9.60	8.80	0.383	4.35	-8.3333	
19.0	18.3	0.957	5.25	-3.9474	



HMX IN WATER SAMPLES

HMX IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative determination of HMX in environmental water samples.

A. TESTED CONCENTRATION RANGE

The tested concentration range in standard and natural water is 0.63 to 12.7 ug/L.

B. SENSITIVITY

The normalized response (integrator counts corrected for attenuation) at the natural water detection limit is 55,000 area counts corresponding to a quantity of 61.3 ng, and 165,000 area counts for 185 ng at the standard water detection limit.

C. DETECTION LIMIT

The detection limits in standard and natural water samples calculated by the Hubaux and Vos procedure are 3.0 ug/L and 0.98 ug/L, respectively.

D. INTERFERENCES

No interferences were encountered in samples of natural water. However, this method may be subject to interferences from neutral, methylene chloride-extractable species which absorb light at 230 nm.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze six extracts in an 8-hour day. One analyst can perform approximately six extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
HMX	Cyclotetramethylenetetranitramine	2691-41-0
	Octahydro-1,3,5,7-tetrazocine	
	1,3,5,7-Tetranitro-1,3,5,7-	
	tetrazacyclooctane	
	Octogen	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

Analyte	Formula	Melting Point (°C)	Boiling Point	Density (g/ml)
нмх	C4H8O8N8	276	-	1.77-1.96*

* There are four polymorphic forms of HMX with this range of densities.

Chemical Structure

C. CHEMICAL REACTIONS

HMX is highly explosive, and caution should be used in handling. HMX is subject to alkaline hydrolysis in aqueous solution.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

- 1. Detector: Perkin-Elmer LC-75 variable-wavelength detector $(\lambda = 230 \text{ nm})$
- 2. Column: Ultrasphere-CN (4.6-mm ID x 25 cm)

Particle size: 5 um

3. Flow Rate/Mobile Phase: 1 ml/min

35% water/65% methanol

- 4. Temperature: 22°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Time: 6.9 minutes

C. HARDWARE/GLASSWARE

- 1. 1-liter separatory touriels (Teflon® or glass) (8).
- 2. 500-ml K-D flasks (8).
- 3. 15-ml K-D receivers (8).
- 4. 3-ball Snyder columns (8).
- 5. 2-ball micro-Snyder columns (8).
- 6. 10-ml graduated centrifuge tubes (8).
- 7. Disposable glass pipette.

D. CHEMICALS

- 1. Nanograde methylene chloride--J.T. Baker Company.
- 2. HPLC-grade acetonitrile--J.T. Baker Company.
- 3. HPLC-grade water--J.T. Baker Company.
- 4. Anhydrous sodium sulfate--reagent grade.
- 5. HPLC-grade methanol.

4. STANDARDS

A. CALIBRATION STANDARDS

- 1. The HMX stock calibration standard (7.9 mg/ml) is prepared by weighing 79.1 mg of HMX in a 10-ml volumetric flask, dissolving the HMX in a few milliliters of acetonitrile (a drop of acetone is added to aid in solubilization), and diluting to the mark with acetonitrile.
- 2. An intermediate stock calibration standard is prepared by pipetting 1 ml of the HMX stock calibration standard into a 25-ml volumetric flask and diluting to the mark with methanol to give a solution containing 316.4 ug/ml of HMX.
- 3. Prepare the working calibration standards by making dilutions of the intermediate stock calibration standard and Working Calibration Standard A with 50% methanol/50% water as follows:

Working Calibration Standard	Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
A	Intermediate Stock	1	100
В	Intermediate Stock	0.5	100
C	Working Standard A	5	25
D	Working Standard A	5	50
E	Working Standard A	5	100

Working Calibration Standard	Concentration (ug/ml)
A	3.16
В	1.58
C	0.63
D	0.32
E	0.16

B. CONTROL SPIKES

1. Prepare the stock control spiking solution (316.4 ug/ml) by diluting 1 ml of the stock calibration standard

(concentration: 7.9 mg/ml) to volume with acetonitrile in a 25-ml volumetric flask.

2. Prepare the working control spike solutions as follows:

Working Control Spike Solution	Solution Used	Volume (m1)	Final Volume (m1)
A	Stock control spike standard	1	50
В	Working Control Spike Standard A	5	50

- 3. Pipet a known amount of the working control spike solutions into standard water. The quantity spiked should be selected to provide a concentration of 0.5 to 10 times the detection limit.
- 4. Determine the accuracy and detection limit for the analyte in standard water by pipetting the working control spike solutions into 500 ml of standard water and analyzing according to the procedure outlined in Section 5.

Working Control Spike Solution	Volume Spiked (ml)	Concentration (ug/L)
		0
В	0.5	0.63
В	1.0	1.27
В	2.0	2.53
A	0.5	6.33
A	1.0	12.7

5. PROCEDURE

A. EXTRACTION

- 1. Measure 500 ml of the water sample into a 1-L separatory funnel.
- Check the pH of the sample with pH paper, and adjust the pH to neutral, if necessary.

- 3. Extract the sample sequentially with three 100-ml portions of methylene chloride. After each portion has been added, shake the funnel vigorously for at least 5 minutes.
- 4. Let the layers separate for about 2 minutes after each extraction.
- 5. Draw off the methylene chloride and pass through a glass funnel containing a small plug of glass wool and about l inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 10-ml K-D receiver.
- 6. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
- 7. Add a boiling chip (Hengar) to the methylene chloride extract in the flask, and attach a 3-ball Snyder column to the apparatus.
- 8. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
- 9. The balls of the Snyder column should chatter actively when the solvent is evaporating.
- 10. When the apparent volume of the solution remaining in the receiver is approximately 1 ml, remove the apparatus from the water bath and allow to cool. After about 1 ml of methylene chloride has drained into the receiver, remove the receiver from the K-D flask.
- 11. Add approximately 2 ml of HPLC-grade methanol to the receiver. Attach a 2-ball micro-Snyder column and reconcentrate. When the apparent volume in the receiver reaches 0.5 ml, remove the receiver from the water bath.
- 12. Repeat Step 11 two times.
- 13. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing quantitatively with HPLC-grade acetonitrile. Raise the extract volume to 1.0 ml in the centrifuge tube with HPLC-grade methanol. Dilute to 2 ml with HPLC-grade water.

- 14. Transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
- 15. The extract is now ready for HPLC analysis.

B. CALIBRATION

- Inject Working Calibration Standards E, D, C, B, and A and a blank singly at the beginning of the analytical run. Inject Working Calibration Standard C at the conclusion of the analytical run to verify constant instrument response.
- 2. Plot the normalized integrator areas versus nanograms injected of each standard to obtain a working curve.

C. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample according to the conditions given in Section 3(B).
- 3. Measure the response of the sample for the HMX peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of HMX according to the following formula:

Concentration (ug/L) =
$$\frac{(A)(V_L)}{V_A}$$

 V_t = Volume of total extract (m1), and

V = Volume of initial sample extracted (L).

7. REFERENCES

None found.

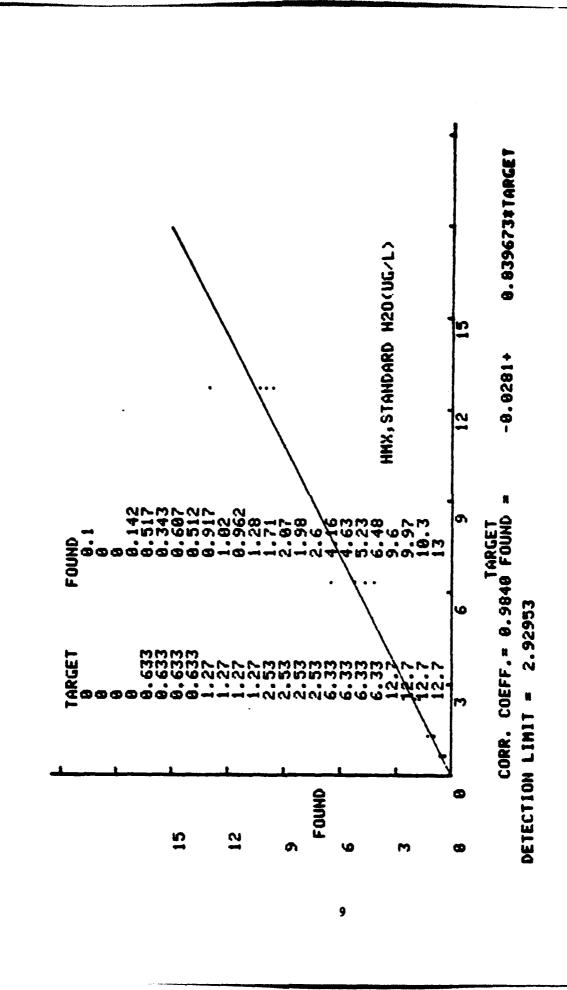
8. DATA

See attached data sheets.

HMX+STANDARD H2C(UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.3000	C • 1 G G O	6.0000	0.0000	0.142	
0.633	0.517	0.343	0.607	0.512	
1.27	0.917	1.02	o•962	1.28	
2.53	1.71	2.07	1.98	2.60	
6.33	4.16	4.63	5.23	6.48	
12.7	9.60	.9.97	10.3	13.0	

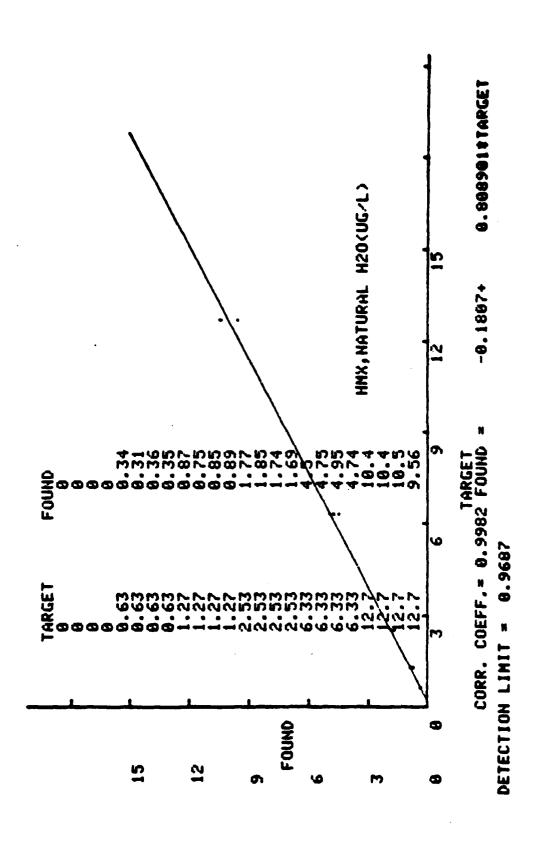
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
5.8956	C.0e95	0.0719	119	0.0000
0.633	0.495	0.115	22.3	-21.6404
1.27	1.04	0.162	15.5	-17.7362
2.53	2.09	0.373	17.8	-17.3913
6.33	5.12	1.60	19.6	-19.0363
12.7	10.7	1.55	14.4	-15.6102

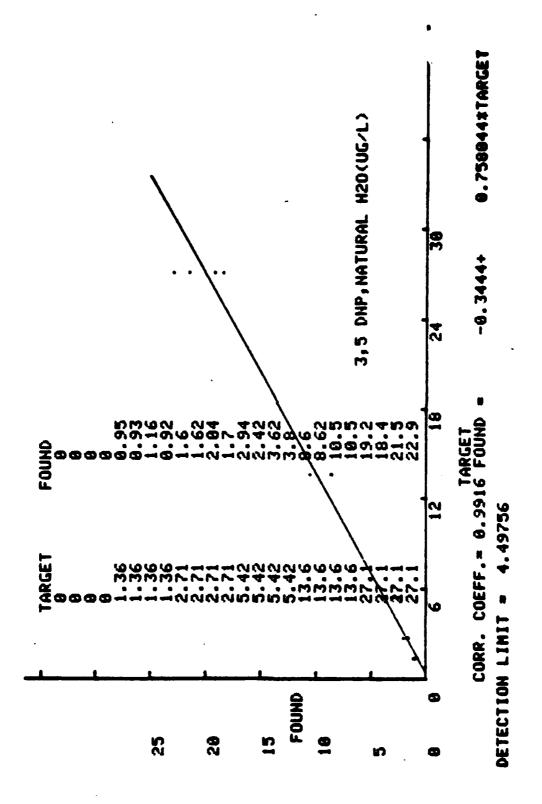


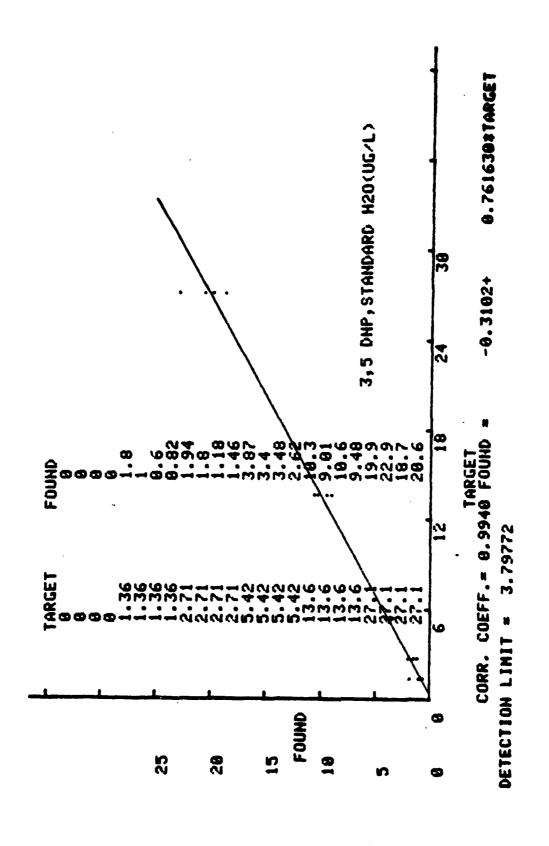
HMX+NATURAL H20(UG/L)

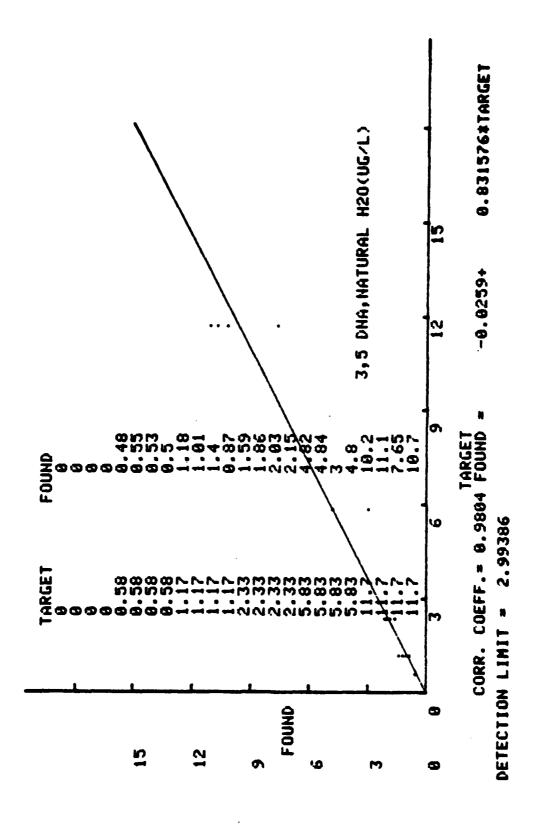
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	0.0000	
€•63€	0.349	0.310	0.360	0.350	
1.27	0.870	0.750	0.850	0.890	
2.53	1.77	1.85	1.74	1.69	
6.33	4.50	4.75	4.95	4.74	
12.7	19.4	10.4	10.5	. 9.56	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
6.3600	0.000)	0.000	0.0000	0.0000
0 • 6 3 0	0.346	0.0216	6.35	-46.C31E
1.27	0840	9.0622	7.40	-33.8583
2.53	1.76	0.0670	3.80	-30.3360
6 • ३३	4.73	0.184	3.89	-25.1975
12.7	16.2	0.43 9	4.30	-19.5669



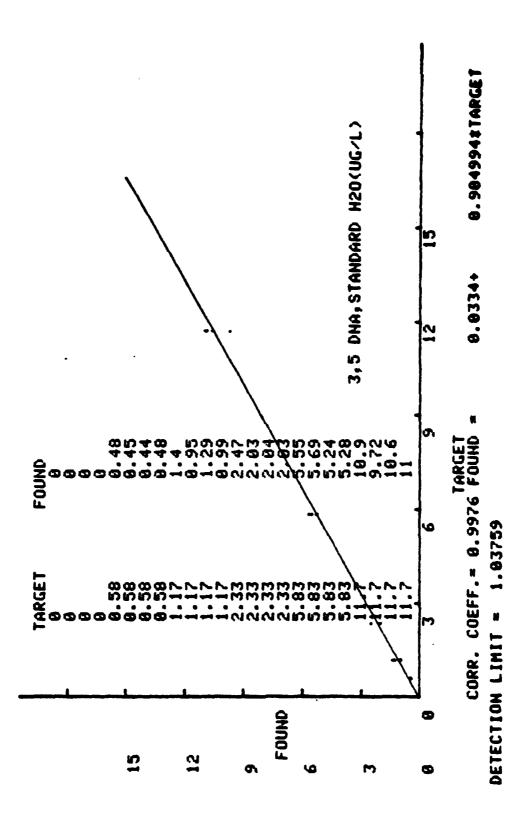


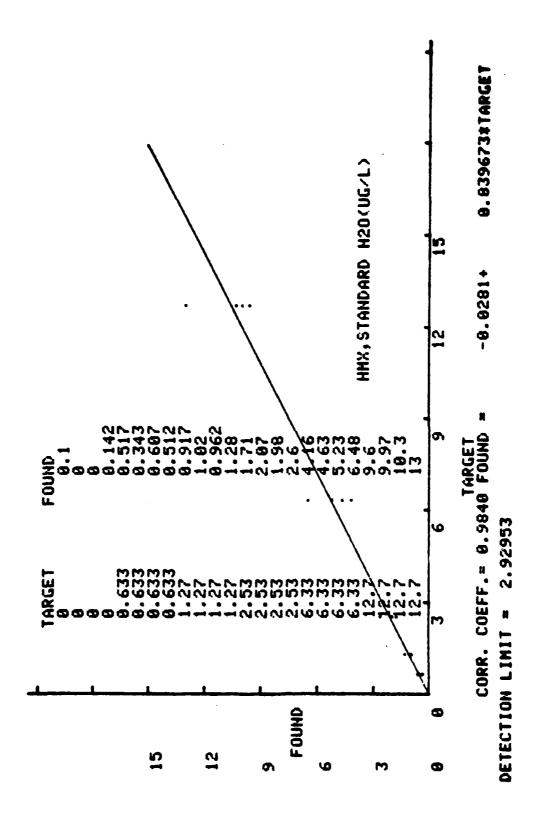


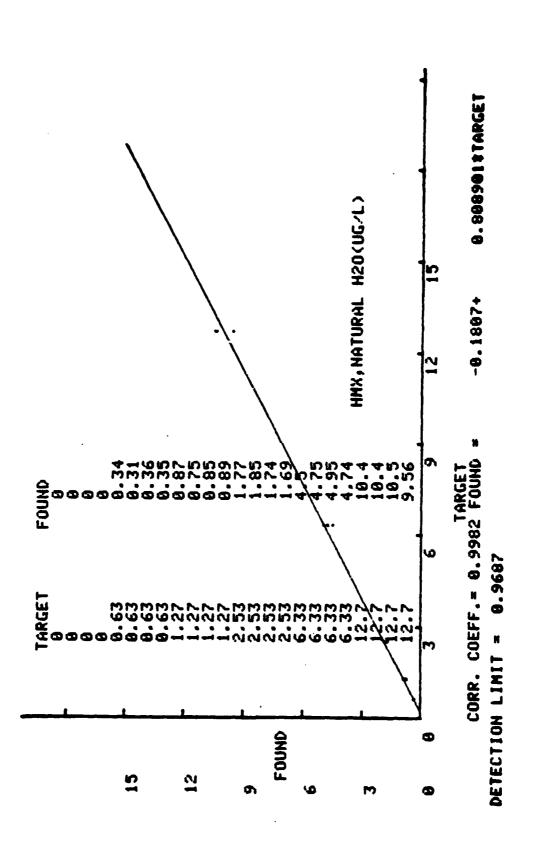


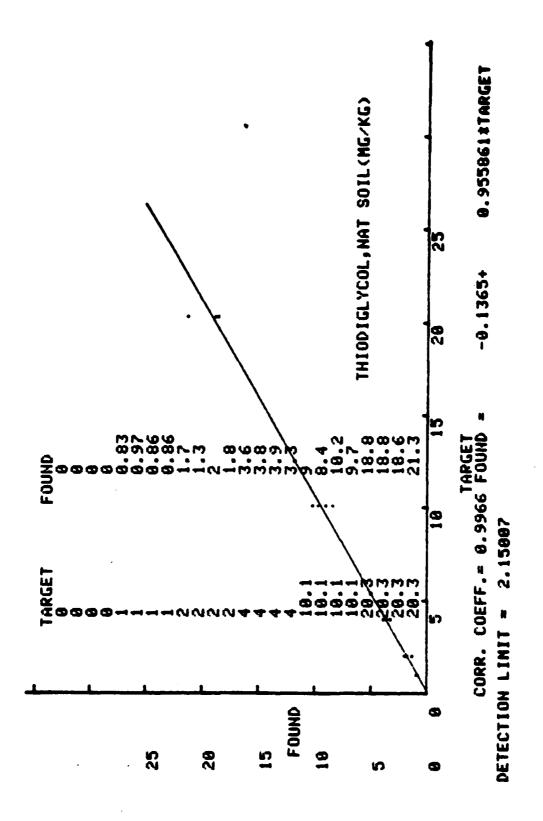
Parameter 1

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HMX IN SOIL SAMPLES

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HMX IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for HMX.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is 0.79 to 15.8 ug/g.

B. SENSITIVITY

The normalized response (integrator count) at the natural soil detection limit, designated in Section 1(C), is listed below:

Integrator Counts	Nanogram
339.000	389

The normalized response (integrator count) at the standard soil detection limit, designated in Section 1(C), is listed below:

Integrator Counts	Nanograms
327,000	375

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is 4.9 ug/g.

The detection limit in standard soil, calculated according to Hubaux and Vos (1970), is 4.7 ug/g.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb UV light at 230 nm and are extractable from soil with methylene chloride/acetone.

Interferences are minimized by silica-gel cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyse 10 extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCIATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
HMX	Cyclotetramethylenetetranitramine	2691-41-0
	Octahydro-1,3,5,7-tetrazocine	
	1,3,5,7-Tetranitro-1,3,5,7-	
	tetrazacyclooctane	
	Octogen	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

		Melting	Boiling	Density
Analyte	Formula	Point (°C)	Point (°C)	(g/ml)
HMX	C/ HgOgNg	276		1.77-1.96*

^{*} There are four polymorphic forms of HMX with this range of densities.

Chemical Structure

C. CHEMICAL REACTION

HMX is highly explosive, and caution should be used in its handling. HMX is subject to alkaline hydrolysis in aqueous solution.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector ($\lambda = 230 \text{ nm}$)

2. Column: Zorbax-CN (4.6-mm ID x 25 cm)

Particle size: 7-8 um

3. Flow Rate/Mobile Phase: 1 ml/min

35% H₂O/65% methanol

4. Temperature: 22°C

5. Injection Volume: 20-ul, fixed loop

6. Retention Time: 6.7 minutes

C. HARDWARE/GLASSWARE

- 1. 50-ml centrifuge tubes with Teflon -lined screw caps (8);
- 2. 500-ml K-D evaporative flasks (8);
- 3. 10-ml graduated K-D receivers (8);
- 4. 3-ball Snyder columns (8);
- 5. 2-ball micro-Snyder columns (8);
- 6. 15-ml graduated centrifuge tubes (8); and
- 7. 10-ml glass or polyethylene syringes with Luer-lock attachments (10).

D. CHEMICALS

- 1. Nanograde methylene chloride;
- 2. HPLC-grade methyl alcohol--J.T. Baker Company;
- 3. HPLC-grade water--J.T. Baker Company;
- 4. Anhydrous sodium sulfate--reagent grade;
- 5. Nanograde hexane;
- 6. Nanograde acetone; and
- 7. Silica-Gel Sep-Paks -- Waters Associates.

4. STANDARDS

A. CALIBRATION STANDARDS

- 1. The HMX stock calibration standard (7.91 mg/ml) is prepared by weighing 79.1 mg of HMX into a 10-ml volumetric flask, dissolving the HMX in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and diluting to the mark with acetonitrile.
- 2. An intermediate HMX stock calibration standard is pregared by pipetting 5 ml of the HMX stock calibration standard into a 50-ml volumetric flask and diluting to the mark with acetonitrile to give a solution containing 791 ug/ml of HMX.
- 3. Prepare a series of working calibration standards by making dilutions of the intermediate calibration standards for HMX.
 Dilute with 50% methanol/50% water as follows:

4

Working Calibration Standard	Standard Diluted	Volume of Standard Used (m1)	Final Volume (ml)
В	HMX (interme	diate) 2	25
C	HMX (interme	diate) 2	50
D	Standard B	5	25
E	Standard B	5	50
F	Standard B	5	100

Working Calibration Standard	Concentration (ug/ml)
В	63.3
C	31.6
D '	12.7
R	6.3
F	3.2

B. CONTROL SPIKES

- 1. Prepare Control Spike Solution A for HMX by pipetting 1 ml of the calibration standard stock for HMX into a 25-ml volumetric flask and diluting to volume with acetone.
- 2. Prepare Control Spike Solution B for HMK by diluting 5 ml of Control Spike Solution A to 50 ml with acetone.

Control	Concentration
Spike Solution	(ug/ml)
A (HMX)	316
B (HMCX)	31.6

3. Allow the soil sample to air dry on the dull side of aluminum foil until it can be sieved through a 30-mesh sieve. (Sediment samples are extracted wet.)

- 4. Weigh 20 g of sieved soil or wet sediment into a 50-ml centrifuge tube with a Teflon lined screw cap.
- 5. Pipette a known amount of the control spike solution for HMX onto the 20-g soil sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

HMX Control Spike Solution	Spike Volume (ml)	Concentration of Spiked Soil (ug/g)
В	0.5	0.79
В	1.0	1.6
В	2.0	3.2
A	0.5	7.9
A	1.0	16

6. Allow the soil to air dry for at least 1 hour. Shake the soil to ensure mixing of the spiking solution throughout the sample.

5. PROCEDURE

A. SAMPLE PREPARATION

- 1. Let all soil samples air dry on the dull side of aluminum foil until they are suitably dry so they can be sieved through a 30-mesh sieve.
- 2. Sample the sieved soil by quartering and weighing 20 g into a 50-ml centrifuge tube.

B. EXTRACTION

- 1. Add 35 ml of 20% acetone in methylene chloride to the centrifuge tube.
- 2. Cap the tube and shake for 3 to 5 minutes.
- 3. Extract the sample sequentially with three 35-ml portions of the methylene chloride/acetone mixture.

- 4. Decant off the methylene chloride/acetone mixture each time, and pass through a glass funnel filled with a small plug of glass wool and approximately 1 inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 10-ml K-D receiver.
- 5. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
- 6. Add a boiling chip (Teflon®) to the methylene chloride extract in the flask, and attach a 3-ball Snyder column to the apparatus.
- 7. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receive: of the K-D nearly up to the joint.
- 8. The balls of the Snyder column should actively chatter when the solvent is evaporating.
- 9. When the apparent volume of the solution remaining in the receiver is about 1 ml, remove the apparatus from the water bath and allow to cool. After approximately 1 ml of solvent has drained back into the receiver, remove the receiver from the K-D flask.
- 10. Add approximately 2 ml of nanograde hexane to the receiver.

 Attach a 2-ball micro-Snyder column and reconcentrate. When
 the apparent volume in the receiver reaches 0.5 ml, remove
 the receiver from the water bath.
- 11. Repeat Step 10 twice.
- 12. Detach the micro-Snyder column from the receiver. With a dispo pipette, transfer the extract into a 10-ml glass syringe fitted with a silica-gel Sep-Pak*. Rinse the receiver three times with 2 ml of 20% methylene chloride in hexane solution, transferring each rinse to the 10-ml syringe. Set aside the receiver for later use.

- 13. Pass the combined rinses through the silica-gel Sep-Pak® at a rate of approximately 1 to 2 ml/min, discarding the eluate.
- 14. Quantitatively rinse the K-D receiver from Step 12 three times with a total of 1 to 2 ml of 50% methanol in methylene chloride solution, transferring each rinse to the 10-ml syringe fitted with the silica-gel Sep-Pak. Add 50% methanol in methylene chloride to the syringe to make a total volume of 10 ml.
- 15. Elute the silica-gel Sep-Pak®, with the 10-ml total volume of 50% methanol in methylene chloride, into another 10-ml K-D receiver at a rate of 1 to 2 ml/min.
- 16. Add a Teflon® boiling chip to eluate, attach a 2-ball micro-Snyder column, and concentrate the sample in a water bath heated to 80°C. When the apparent volume of the solution is about 0.5 ml, remove the apparatus from the water bath.
- 17. Detach the micro-Synder, and add approximately 2 ml of HPLC methanol to the receiver. Reconcentrate the sample to 0.5 ml.
- 18. Repeat Step 17 twice.
- 19. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube rinsing quantitatively with HPLC methanol. Raise the extract volume to exactly 2.5 ml in the centrifuge tube with HPLC methanol. Dilute to 5 ml with HPLC water.
- 20. Transfer to a 5-ml amber, septum-sealed vial for storage at
- 21. The extract is now ready for chromatography by HPLC.

C. ANALYSIS

- 1. Inject 20 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample using the conditions given in Section 3(B).
- 3. Measure the response of the component of interest.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard-versus-peak area counts.
- B. Determine the concentration of HMX according to the following formula:

Concentration (ug/g) =
$$\frac{(A)(V_t)}{W_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve,

V_t = Volume of total extract (ml), and W_s = Weight of initial sample extracted (g).

- 7. REFERENCES
 None found.
- 8. DATA
 See attached data sheets.

HMX.STANDARD SOIL (UG/G)

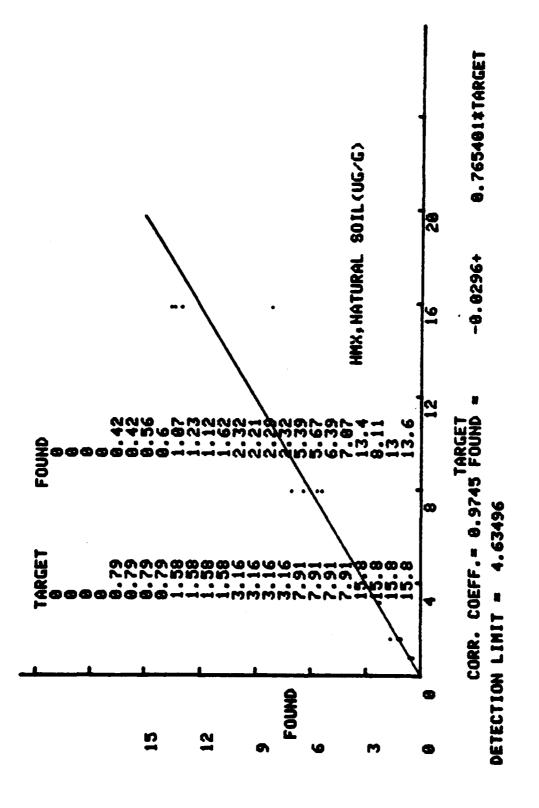
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0530	0.0000	0.0000	0.0000	
0.790	0.620	0.660	0.680	0.850	
1.58	1.37	1.41	1.64	1.03	
3.16	3.30	3.06	3.22	2.32	
7.91	6.16	6.03	4.90	6.91	
15.8	13.1	13.5	8.32	10.9	

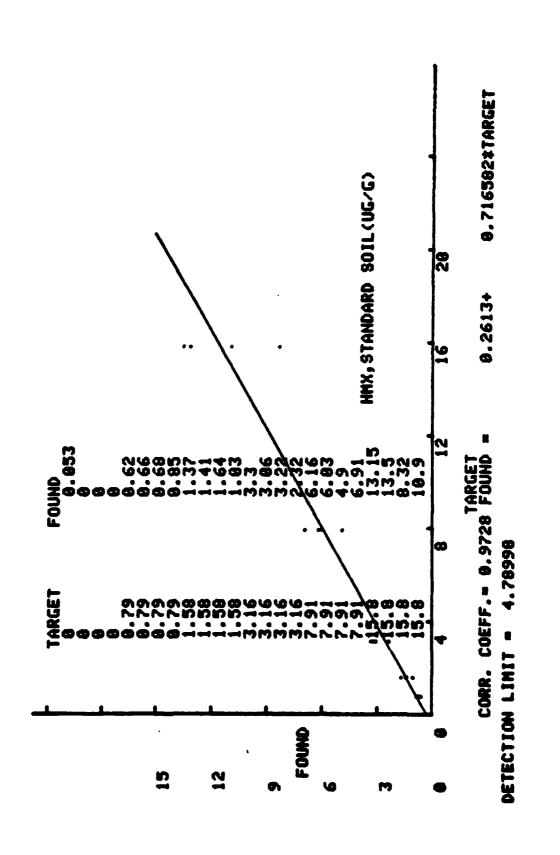
TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0132	0.0265	200	0.0000
0.790	0.702	0.101	14.4	-11.0760
1.58	1.36	0.252	18.5	-13.7658
3.16	2.97	0.448	15.1	-5.8544
7.91	6-00	0.830	13.8	-24.1467
15.8	11.5	2.39	20.9	-27.4209

HMX.NATURAL SOIL(UG/G)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0093	0.0000	0.0000	0.0000	0.0000	
0.790	0.420	0.420	0.560	0.600	
1.58	1.07	1.23	1.12	1.62	
3.16	2.32	2.21	2.29	2.32	
7.91	5.39	5.67	6.39	7.07	
15.8	13.4	8.11	13.0	13.6	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.000	0.0000	0.000.0
0.790	0.500	0.0938	18.8	-36.7089
1.58	1.26	0.249	19.8	-20.2532
3.16	2.28	0.0520	2.27	-27.6899
7.91	6.13	0.755	12.3	-22.5032
15.8	12.0	2.62	21.8	-23.8766





DPA IN SOIL AND SEDIMENT SAMPLES

DPA IN SOIL AND SEDIMENT SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of DPA in environmental soil and sediment samples.

This method was developed and tested in two soil matrices: a red clay obtained from the Alabama Army Ammunition Plant and a topsoil from North Central Florida. These two soils are subsequently referred to as the standard soil and the natural soil, respectively.

A. TESTED CONCENTRATION RANGE

The tested concentration ranges are:

Matrix	Tested Concentration Range (ug/g)
Standard Soil	1.3 to 26
Natural Soil	1.5 to 30

B. SENSITIVITY

The normalized response (integrator counts x attenuation at the documented detection limits designated in Part C below are:

Matrix	Integrator Counts	Quantity (picograms)	
Standard Soil	1,221	1,500	
Natural Soil	1,627	1,600	

C. DETECTION LIMITS

The detection limits, calculated according to Hubaux and Vos (1970), are:

Matrix	Concentration (ug/g)
Standard Soil	1.5
Natural Soil	1.6

D. INTERFERENCES

This method contains no cleanup step and thus may be subject to interferences from nitrogen- or phosphorus-containing compounds which co-elute with the chromatographic peak for DPA. The detection method used for this work is, however, very selective for nitrogen (and phosphorus), with a selectivity ratio of 1,000:1 for nitrogen versus carbon. This method has been tested on a variety of soil matrices with no apparent interferences.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 8 extracts in an 8-hour day. One analyst can perform approximately 10 extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCIATURE AND CHEMICAL ABSTRACT SERVICE (CAS)
REGISTRY NUMBER

	Alternate	CAS Registry	
Analyte	Nomenclature	Number	
DPA		122-39-4	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTES

		Melting	Boiling	Density
Analyte	<u>Pormula</u>	Point (°C)	Point (°C)	(g/ml at 20°C)
DPA	C12H11N	52-54	302	1.16

C. CHEMICAL REACTIONS None found.

3. APPARATUS

A. INSTRUMENTATION

Perkin Elmer Sigma 2 GC interfaced to a Spectra Physics 4100 Computing Integrator.

B. GC PARAMETERS

- 1. Detector: Nitrogen-phosphorus specific; bead current setting--6.8 to 7.0.
- Column: 1/4-inch x 2-mm ID x 10-ft glass
 Packing: 1.5% OV-17 on 80/100 mesh supelcoport
- 3. Gas Flow

Carrier: Nitrogen (50 ml/minute)

Detector: Hydrogen (1-2 ml/minute)

Air (100 ml/minute)

4. Temperature

Injector: 250°C Detector: 300°C

Oven Temperature: Isothermal at 160°C

- 5. Injection Volume: 5 ul
- 6. Retention Time: 4.0 minutes

C. HARDWARE/GLASSWARE

- 1. 50-ml, Teflone-lined capped centrifuge tube.
- 2. Glass disposable pipettes.
- 3. Centrifuge, capable of handling 50-ml centrifuge tubes.
- 4. Class A volumetric flasks: 50 ml, 10 ml.
- 5. Metal spatula.

D. CHEMICALS AND REAGENTS

- 1. Toluene, nanograde, distilled in glass.
- 2. Methanol, nanograde, distilled in glass.
- 3. 0.01N sodium hydroxide solution.
- 4. Analytical standards (SARMs or equivalent).

4. STANDARDS

A. CALIBRATION STANDARDS

 Prepare a calibration standard stock (11.7 mg/ml) by weighing 117 mg of the DPA SARM in a single 10-ml volumetric flask.

Dissolve in a few ml of toluene and dilute to volume with toluene. Wrap the flask in foil and store in a freezer. No evidence of degradation was observed over a 6-month period.

- 2. Prepare the dilute stock calibration standard by pipetting 1 ml of the calibration standard stock and diluting to volume with toluene in a 100-ml volumetric flask. Label this solution and store in an amber, septum-sealed vial at 4°C.
- Prepare the working stock calibration standards by making dilutions with toluene of dilute stock calibration standard as follows.

Working Stock Calibration Standard	Dilute Stock Calibration Standard	Final Volume (ml)	Concentra- tion (ng/g)
A	100 ul	50	234
В	200 ul	50	468
C	400 ul	50	937
D	1.0 =1	50	2,340
E	2.0 =1	50	4,680
F	4.0 ml	50	9,360
G	10.0 ml	50	23,400

Store standards in amber septum-sealed vials at 4°C until ready to use.

A

B. CONTROL SPIKES

 Prepare the stock control spike solution (2.57 mg/ml) by weighing approximately 25.7 mg of the DPA SARM into a single 10-ml volumetric flask.

Dissolve in a few ml of methanol and dilute to volume with methanol.

- 2. Dilute the stock control spike solution by pipetting 1 ml into a 10-ml volumetric flask and diluting to volume with methanol to obtain the working stock control spike solution.
- 3. Pipet a known amount of the working control spike into a standard soil sample. The quantity spiked should be selected to approximately double the expected concentration or to provide a concentration of 0.5 to 10 times the detection limit. The precision, accuracy, and detection limits were determined by spiking 10-g samples of soil at the following levels:

Volume	Concentration	
Spiked (ul)	(ug/g)	
0	0	
50	1.28	
100	2.57	
200	5.14	
500	12.8	
1,000	25.7	

- 4. After spiking the soil, enough toluene is added to just wet the sample. The mixture is allowed to air-dry for 1 hour before analysis.
- 5. Perform the procedures in Section 5, starting with Step B2.

5. PROCEDURE

A. SAMPLE PREPARATION

- 1. Let all soil samples air-dry on the dull side of aluminum foil until they can be sieved through a 30-mesh sieve.
- 2. Sediment samples are extracted wet.
- 3. Determine the dry weight of the sediment or soil by weighing 5 g of sample into a tared beaker. Dry the soil at 105°C until constant weight. Record the dry weight. Calculate the percent moisture.

B. EXTRACTION

- 1. Weigh 10 g of soil or 15 g of wet sediment (to the nearest 0.1 g) into a 50-ml centrifuge tube.
- 2. Add 20 ml of 0.01N sodium hydroxide, 10 ml of toluene, and 5 ml of methanol to the tube and cap. Shake the tube vigorously for 3 minutes.
- 3. Place the tube in the centrifuge, and centrifuge at medium speed ("5") until the solids separate from the solvent (10 minutes).
- 4. Withdraw the toluene/methanol layer (top layer) using a glass pipet and transfer to a 50-ml volumetric flask.
- 5. Repeat the extraction with 10 ml of toluene and 5 ml of methanol two more times.
- 6. Combine the toluene extracts in the 50-ml volumetric flask.
- 7. Dilute the extract with toluene to the 50-ml mark.

8. Shake the flask and transfer approximately 1 ml to a septum-sealed vial. Cap securely with septum and seal. Protect from light by wrapping the sample vial with foil.

C. CALIBRATION

- Inject the working standards singly at the beginning of the analytical run and one standard at the conclusion of the analytical run.
- 2. Plot the normalized peak height versus micrograms injected of each standard to obtain a working curve.
- D. ANALYSIS

Inject 5 ul of the toluene extract onto the GC column.

6. CALCULATIONS

A. Determine the concentration of the analyte according to the following formula:

Concentration (ug/g) =
$$\frac{(A)(V_t)}{(V_i)(W)}$$

where A = Amount of analyte found (ug), determined from the standard curve,

Vt = Volume of total extract (ml),

Vi = Volume of extract injected (ml), and

W = Sample weight (g).

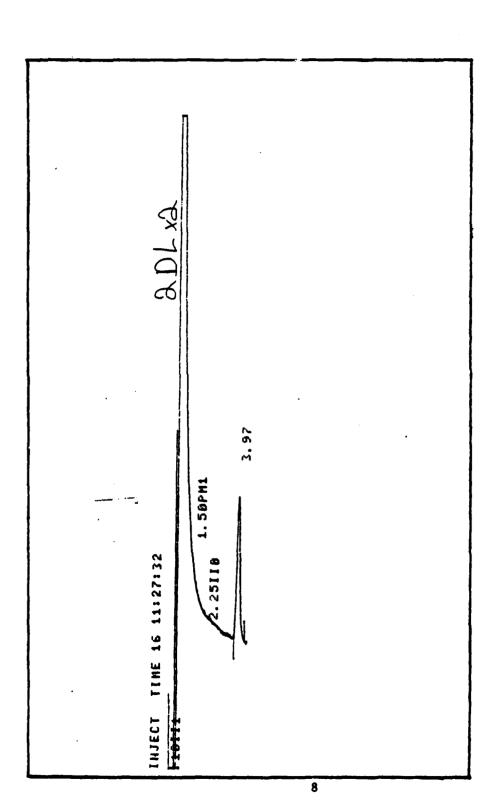
- B. Correct for percent moisture in the original soil/sediment sample.
- 7. REFERENCES

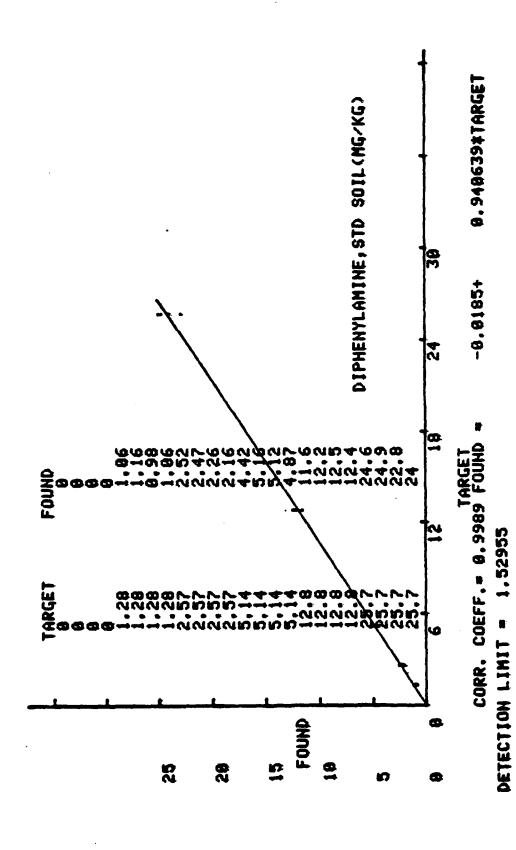
None found.

8. DATA

See attached data sheets.

Figure 1 Chromatogram of Diphenylamine (2340 pg injected).

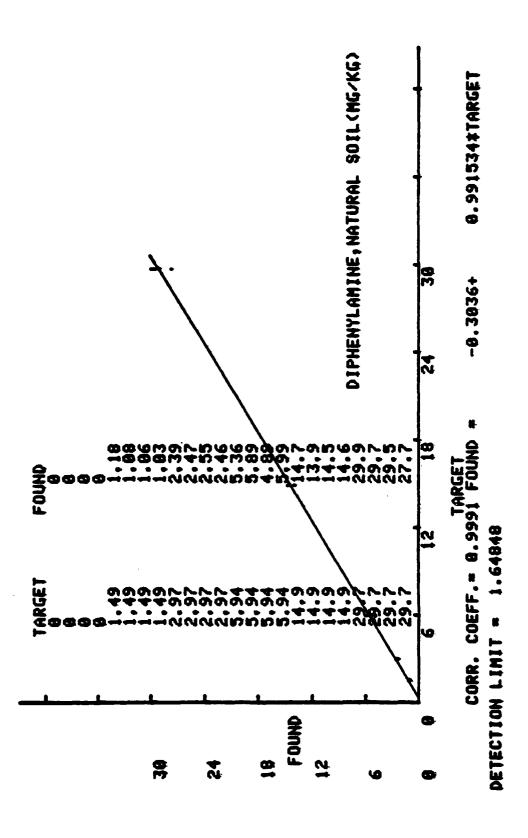




DIPHENYLAMINE, STD SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.3000	0.0000	0.0000	
1.28	1.06	1.16	0.980	1.06	
2.57	2.52	2.47	2.26	2•16	
5.14	4.42	5.16	5.12	4.87	
12.8	11.6	12.2	12.5	12.4	
25.7	24•6	24.9	22.8	24.0	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.3025	0.0000	0.000	0.000	0.0000	
1.28	1.06	3.0737	6.92	-16.7969	
2.57	2.35	0.171	7.26	-8-4650	•
5 • 1 4	4.89	6.340	6.95	-4.8152	
12.8	12.2	0.403	3.31	-4.5828	
25.7	24.1	0.929	3.86	-6.3230	,



.1

DIPHENYLAMINE.NATURAL SOIL (MG/KG)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	. 0.0000	0.0000	0.0000	0.0000	
1.49	1.18	1.08	1.06	1.03	
2.97	2.39	2.47	2.55	2.46	
5.94	5.36	5.89	4.89	5.99	
14.9	14.7	13.9	14.5	14.6	
29.7	29.9	29.7	29.5	27.7	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT 1MPRECISION	PERCENT INACCURACY	
0.0000	0.0003	6 • d 0 0 0	0.0000	0.0000	
1.45	1.09	0.0650	5.98	-27.0134	
2.97	2.47	0.0655	2.66	-16.9192	
5.94	5.53	0.510	9.21	-6.8633	
14.9	14-4	0.359	2.49	-3.1879	
29.7	29•2	1.01	3.47	-1.6835	

UDMH IN WATER SAMPLES

UDMH IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for UDMH.

A. TESTED CONCENTRATION RANGE

The tested concentration range for standard and natural water is 5 to 107 ug/L.

B. SENSITIVITY

The normalized responses (peak heights) at the detection limits designated in Section C (below) are 325 mm for 1.36 ng in natural water and 423 mm for 2.01 ng in standard water.

C. DETECTION LIMITS

The detection limits, calculated according to Hubaux and Vos (1970), are 11 ug/L for natural water and 16 ug/L for standard water.

D. INTERFERENCES

This method may be subject to interferences from compounds which can be readily oxidized under an electrochemical potential of +0.9 volt. Phenolic compounds are included in this class; however, chromatographic conditions were selected to minimize interferences from the commonly found priority pollutant phenols.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyse 10 samples in an 8-hour day.

2. CHEMISTRY

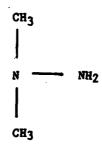
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number	
UDMH	asym-dimethylhydrazine		
	l,l-dimethylhydrazine	57-14-7	
	unsym-dimethylhydrazine		
	N,N-dimethylhydrazine		
	Dimazine		

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

	Molecular	Melting	Boiling	Density
Analyte	Formula	Point	Point	(g/m1)
UDMH	C ₂ H ₈ N ₂	-58°C	63.9°C	0.791

Chemical Structure



C. CHEMICAL REACTIONS

UDMH is a powerful reducing agent used as the base in rocket fuel formulations. UDMH is highly corrosive and irritating to skin, eyes, and mucous membranes. UDMH is readily oxidized in alkaline solution by a number of oxidants (HgO, halogens, and halates) to produce tetrazine. Tetrazines are inherently unstable and split out N_2 under thermal or protolytic conditions. In acidic solutions, UDMH reacts to form diagenium salts, $(CH_3)_2N^+ = NH X^-$, which react as dienophiles with

conjugated dienes in the Diels-Alder reaction. Hydrazines reduce many commonly found metal ions to lower valence states or to the metals themselves. Over 23 metal ions have been shown to react with hydrazines.

The kinetics of the decomposition of UDMH in aqueous solutions were briefly examined, and it has been found that the halflife for the disappearance of UDMH is less than 1 day. For this 'reason, standards for the UDMH analysis must be prepared fresh daily, immediately before the analysis is begun. Samples should be analyzed as soon as possible after collection. Chromatograms which illustrate the decomposition of UDMH in water are presented in Figure 1.

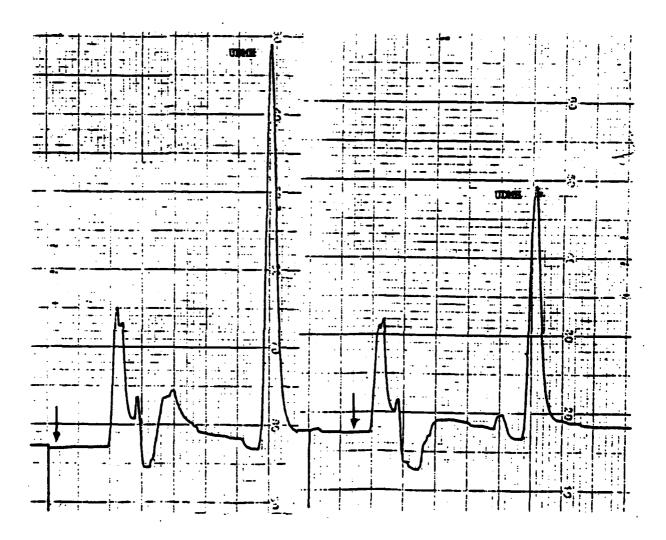
3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Bioanalytical Systems Model EC-2A Electrochemical Detector interfaced to a linear strip-chart recorder.

B. HPLC INSTRUMENTAL PARAMETERS

- Detector: Bioanalytical Systems, Inc. Model EC-2A
 Electrochemical Detector with a glassy carbon electrode
 Potential = +0.9 volt
- 2. Column: Zorbax C-8 (4.6-mm ID x 25 cm)
 Particle size = 5-6 um
- 3. Flow Rate/Mobile Phase: 1 ml/min/50% acetonitrile/50% 0.09 M PO $_{\Delta}^{2-}$ buffered to pH = 7
- 4. Temperature: 22°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Time: 7.1 minutes



Chromatograms of UDMH at 50 ug/L in Natural Surface Water Time = 0 hours on left and 24 hours on right (pH = 6.0) Figure 1.

C. HARDWARE/GLASSWARE

- 1. 25-ml volumetric flask (1)
- 2. 50-ml volumetric flask (10)

D. CHEMICALS

- 1. HPLC-grade acetonitrile, J.T. Baker Company.
- 2. HPLC-grade water, J.T. Baker Company.
- 3. Potassium dihydrogenphosphate, J.T. Baker Company.
- 4. Potassium monohydrogenphosphate, J.T. Baker Company.

4. STANDARDS

A. CALIBRATION STANDARDS

- Prepare a stock calibration standard (6.72 mg/ml) by weighing 168 mg of the UDMH SARM in a 25-ml volumetric flask.
- 2. Dissolve the UDMH in a few ml of HPLC-grade acetonitrile, and dilute to volume with acetonitrile. Wrap the flask in foil, and store at 4°C.
- 3. Prepare Intermediate Stock Calibration Standard A by pipetting 1 ml of the stock calibration standard into a 50-ml volumetric flask and diluting to volume with acetonitrile. Transfer the solution to an amber, septum-sealed vial, and store at 4°C.
- 4. Prepare Intermediate Stock Calibration Standard B by pipetting 1 ml of Intermediate Stock Calibration Standard A into a 50-ml volumetric flask and diluting to volume with acetonitrile. Transfer this solution to an amber, septum-sealed vial, and store at 4°C.
- 5. Prepare a series of working calibration standards by making dilutions with 50% acetonitrile/50% 0.09 M phosphatebuffered water (pH = 7) as follows:

Working Calibration Standard	Concentration (ng/g)	Standard Diluted	Volume of Standard Used (ml)	Final Volume (m1)
C	54	Intermediate Stock B	1	50
D	27	Intermediate Stock B	0.5	50
E	10.8	Working Standard C	10	50
F	5.4	Working Standard D	10	50
G	2.7	Working Standard D	5	50

The standards must be prepared fresh daily.

B. CONTROL SPIKES

- Pipet a known amount of Intermediate Stock Calibration Standard B into 25 ml of standard or natural water samples, and analyze by the procedure outlined in Section 5 below. The quantity spiked should be selected to provide a concentration of 0.5 to 10 times the detection limit.
- 2. Determine the precision, accuracy, and detection limit for the analyte.

Concentration (ng/g)	Volume (ml) of Standard B Spiked into 25 ml		
	0.0		
5.36	0.050		
10.7	0.100		
21.4	0.200		
53.6	0.500		
107.2	1.00		

5. PROCEDURE

A. SAMPLE PREPARATION

- 1. Pipet 25 ml of the water sample into a 50-ml volumetric flask.
- Add approximately 15 ml of HPLC-grade acetonitrile to the volumetric flask, shake until thoroughly mixed, and wait until all the gas bubbles formed on mixing disappear (approximately 5 minutes).

- 3. Dilute to the 50-ml mark with HPLC-grade acetonitrile.
- 4. The sample is now ready for analysis by HPLC.

B. CALIBRATION

- Inject 250 ul of each of the working calibration standards and a blank singly at the beginning of the analytical run. Inject Working Calibration Standard E at the end of the analytical run to verify constancy of the instrument response.
- Plot the normalized peak height (mm) versus the concentration for each standard to obtain a working calibration curve.

C. ANALYSIS

- 1. Inject 250 ul of the sample onto the LC column.
- 2. Perform analysis of the sample according to the conditions given in Section 38.
- 3. Measure the peak height (mm) for the UDMH peak.

6. CALCULATIONS

Calculate the concentration of UDMH according to the following equation:

Concentration ug/L = 2A/1000

where: A is the concentration (ug/ml) obtained from the calibration curve.

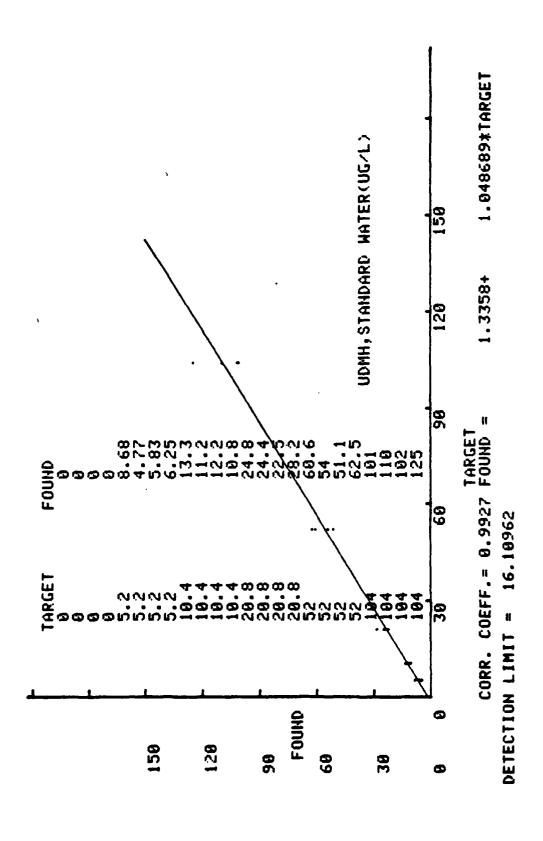
The injection volume is 250 ul, and the factor of 2 is due to the dilution of the sample with acetonitrile. This dilution is necessary to match sample and mobile phase compositions to minimize baseline disturbances arising from the sample injection.

7. REFERENCES

None found.

8. DATA

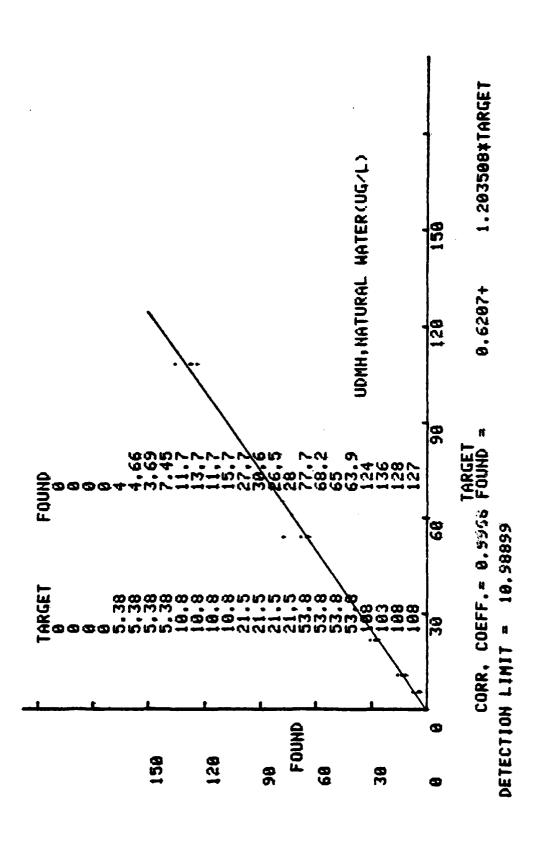
See attached data sheets. The target values reported are the averages of the values obtained over the 4 successive days of spiking experiments. Since UDMH decomposes rapidly, the standards were prepared fresh each day, and exact duplication of the target values was not practically feasible. The actual target values were closely spaced about the averages with a relative standard deviation of 2 percent.



UDMH.STANDARD WATER(UG/L)

TAFGET CONCENTRATION	1	DAY	3	4	
0.0000	0.0000	C • 0 0 0 0	0.0000	C.00G0	
5.20	8.68	4.77	5.63	6.25	
10.4	13.3	11.2	12.2	10.8	
20.8	24.8	24.4	22.5	28.2	
52.0	60.6	54.0	51.1	62.5	
104	181	110	102	125	_

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
5 • 2 0	6.38	1.65	25.9	22.7
10.4	11.9	1.12	9.41	14.2
20.8	25.0	2.37	9.50	20.1
52.0	57.0	5.39	9.44	9.71
104	110	11.1	10.1	5 • 29



UDMH+NATURAL WATER (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4	
C • C O O G	0.0000	8.0000	0.0000	0.0000	
5.38	4.00	4.66	3.69	7.45	
10.8	11.7	13.7	11.7	15.7	
21.5	27.7	30.6	26.5	28 • G	
53 • 8	77.7	68•2	65.0	63.9	
108	124	136	128	127	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0.0000	0.0000	0.0000	0.0000	
5.38	4.95	1.72	34.6	-7.9926	
10.8	13.2	1.91	14.5	22.2	
21.5	28.2	1.73	6.12	31.2	
53.8	68.7	6.27	9.13	27.7	
108	129	5.12	3.98	19.2	

ATNBA IN WATER SAMPLES

ATNBA IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for ATNBA.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard water is listed below in ug/L.

 Analyte
 Range (ug/L)

 ATNBA
 0.59 to 11.8

B. SENSITIVITY

The normalized response (integrator counts) at the natural water detection limit designated in Section 1C is listed below:

Analyte Integrator Counts Nanograms
ATNBA 107,111 248

The normalized response (integrator counts) at the standard water detection limit designated in Section 1C is listed below:

 Analyte
 Integrator Counts
 Nanograms

 ATNBA
 160,343
 371

C. DETECTION LIMIT

The detection limit in natural water, calculated according to Hubaux and Vos (1970), is listed below:

Analyte Detection Limit (ug/L)
ATNBA 2.2

The detection limit in standard water, calculated according to Hubaux and Vos (1970), is listed below:

Analyte

Detection Limit (ug/L)

ATNBA

3.3

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb light at 254 nanometers and are extractable from water at pH = 5 with methylene chloride/acetone. Interferences are minimized by Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 8 extracts in an 8-hour day. One analyst can perform approximately 8 extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS)
REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
ATNBA	2,4,6-trinitrobenzene carbonal	606-34-8
	2,4,6-trinitrobenzene carbox-	
	aldehyde	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

		Melting	Boiling	Density
Analyte	Formula	Point (°C)	Point (°C)	(g/ml)
ATNBA	C7 H2O7N2	119		

Chemical Structure

C. CHEMICAL REACTIONS

ATNBA is explosive and rapidly decomposes in light; it must be protected by use of amber glass or aluminum foil. It decomposes rapidly to 135TNB in basic solution via a red-colored intermediate. It is slowly decomposed to 135TNB by dissolved oxygen, water, or upon heating with water or alcohol.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with an Altex Model 153 UV detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Altex Model 153 UV detector ($\lambda = 254 \text{ nm}$).

2. Column: Ultrasphere C18 (4.6-mm ID x 25 cm)

Particle size: 5 um

3

3. Flow Rate/Mobile Phase: 1 ml/min

45% H₂O/55% methanol

- 4. Temperature: 25°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Times:

Analyte	Retention Time (minutes	<u>1)</u>
ATNBA	5.8	
135TNB (decomposition product)	6.4	

C. HARDWARE/GLASSWARE

- 1. 1-L Teflon separatory funnels with screw caps (8);
- 2. 250-ml flat-bottomed boiling flasks (8);
- 3. Glass filter funnels (8);
- Rotary evaporation with controlled-temperature water baths
 (4);
- 5. Cold trap and vacuum pump;
- 6. 15-ml graduated centrifuge tubes (8); and
- 7. 5-ml glass syringes with Luer-lock attachments (8).

D. CHEMICALS

- 1. Nanograde methylene chloride;
- 2. HPLC-grade methyl alcohol--J.T. Baker Company;
- 3. HPLC-grade water--J.T. Baker Company;
- 4. Reagent-grade anhydrous sodium sulfate;
- 5. Nanograde pentane;
- 6. HPLC-grade acetonitrile--J.T. Baker Company;
- 7. Florisil® Sep-Paks®--Waters Associates;
- 8. 85% phosphoric acid--ACS;
- Colorphast[®] indicator sticks (MCB Manufacturing Chemists, Inc.); and
- 10. Pasteur pipettes.

4. STANDARDS

A. CALIBRATION STANDARDS

- 1. The ATNBA stock calibration standard (1.06 mg/ml) is prepared by weighing 10.6 mg of ATNBA into a 10-ml volumetric flask, dissolving the ATNBA in a few ml of acetonitrile, and diluting to the mark. The stock solution should be stored under nitrogen, in an amber vial in a freezer. It should be replaced on a monthly basis or sooner if degradation is observed (bright pink color or appearance of 135TNB on chromatogram).
- 2. An intermediate calibration standard is prepared by diluting l ml of the stock solution to 100 ml with acetonitrile that has been vacuum degassed. This standard should be stored under nitrogen, in an amber vial, in the freezer. It should be replaced on a weekly basis, or sooner if degradation is observed.
- 3. Prepare a series of working calibration standards by making dilutions of the intermediate calibration standard. The standards should be made fresh just prior to analysis.

 Dilute with a 50% acetonitrile/50% water solution (that has been vacuum degassed and kept under nitrogen) as follows:

Working Calibration Standard	Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
A	Intermediate stock	5.0	10.0
В	Intermediate stock	2.0	10.0
C	Intermediate stock	1.0	10.0
D	Intermediate stock	0.50	10.0
E	Intermediate stock	0.20	10.0
F	Intermediate stock	0.10	10.0

Working Calibration Standard	Concentration (ug/ml)
A	5.30
В	2.12
С	1.06
D	0.530
E	0.212
F	0,106

B. CONTROL SPIKES

- 1. Use the intermediate calibration standard as the control spiking solution.
- 2. Measure out 900 ml of water into a 1-L separatory funnel.
- 3. Pipet a known amount of the control spike solution into the sample and mix thoroughly. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

Spike Volume (m1)	Concentration of Spiked Water (ug/L)
0.05	0.59
0.1	1.18
0.2	2.36
0.5	5.89
1.0	11.78

- 4. Adjust the pH of the sample to 5 with 85% H₃PO₄.
- 5. Extract the samples according to the procedure presented in Section 5B.

5. PROCEDURE

A. SAMPLE PREPARATION

- 1. Measure 900 ml of the sample into a 1-L Teflon separatory funnel.
- 2. Adjust the sample pH to 3 with 85% H₃PO₄ by dropwise addition.

B. EXTRACTION

- 1. Add 60 ml of methylene chloride to the separatory funnel.
- 2. Extract the sample by shaking the funnel for 3 minutes and then allowing 10 minutes phase separation time. Emulsions formed should be separated by centrifuging or sonication.
- 3. Drain the methylene chloride through a glass funnel filled with a small plug of glass wool and approximately 20 g of anhydrous sodium sulfate into a 250-ml boiling flask.
- 4. Repeat Steps 1 through 3 twice more and then combine extracts. The extracts should be protected from light by covering the boiling flask with aluminum foil.
- 5. After the third extract has been transferred to the flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
- 6. Concentrate the methylene chloride extract by rotary evaporation on a 35°C water bath.
- 7. When the apparent volume of the solution remaining in the flask is about 1 ml, remove the apparatus from the rotovap. Using a Pasteur pipette, transfer the extract to a 15-ml graduated centrifuge tube. Rinse the flask with an additional 1 ml of methylene chloride. Transfer the rinse to the centrifuge tube and raise the volume to 2.5 ml.
- Raise to a final volume of 5 ml by adding nanograde pentane.
 Mix.
- Equip a 5-ml syringe with a Florisil® Sep-Pak®. Pour the extract into the syringe, and pass through the Sep-Pak®, discarding the eluent.

- 10. Measure 5 ml of a 20% acetonitrile/80% methylene chloride solution in the centrifuge tube, and pour into the syringe. Elute the Sep-Pak® into the original boiling flask.
- 11. Evaporate the extract on the rotovap to an approximate volume of 1 ml. Add 10 ml of acetonitrile, and reduce the volume to 1 ml. Add a further 10 ml of acetonitrile, and reduce the extract to a final volume of 0.5 ml.
- 12. Transfer the 0.5 ml of extract to a 15-ml graduated centrifuge tube using a Pasteur pipette. Rinse the flask with an additional 0.5 ml of acetonitrile (which has been vacuum degassed and stored under nitrogen) and transfer to the centrifuge tube. Raise to a final volume of 2.0 ml with HPLC-grade water (vacuum degassed and stored under nitrogen).
- 13. Transfer to a 3-ml amber, septum-sealed vial for storage at 4°C.
- 14. The extract is now ready for chromatography by HPLC. The extract must be analyzed within 24 hours of time of extraction.

C. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample using the conditions given in Section 3B.
- 3. Measure the response of the component of interest.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of ATNBA according to the following formula:

Concentration (ug/L) =
$$\frac{(A)(V_t)}{V_s}$$

 V_t = Volume of total extract (ml), and

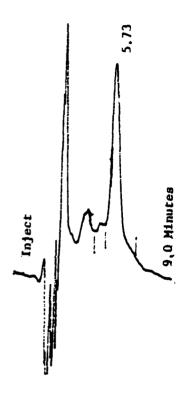
 V_s = Volume of initial sample extracted (L).

7. REFERENCES

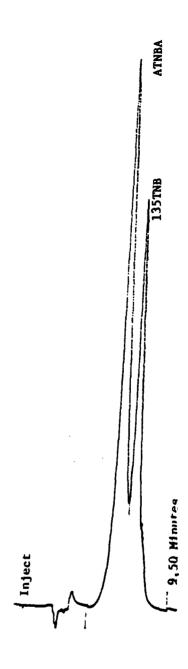
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8. DATA

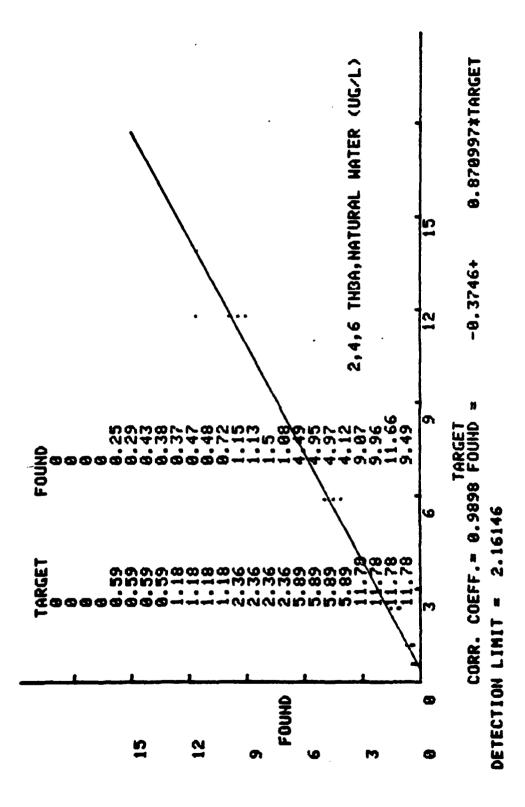
See attached data sheets.



Chromatogram of a Natural Water Extract Spiked at the 2.36-ug/L Level with ATNBA



C.romatogram of ATNBA and 135TNB (Decomposition Product)
Under the Conditions Given in Section 3B

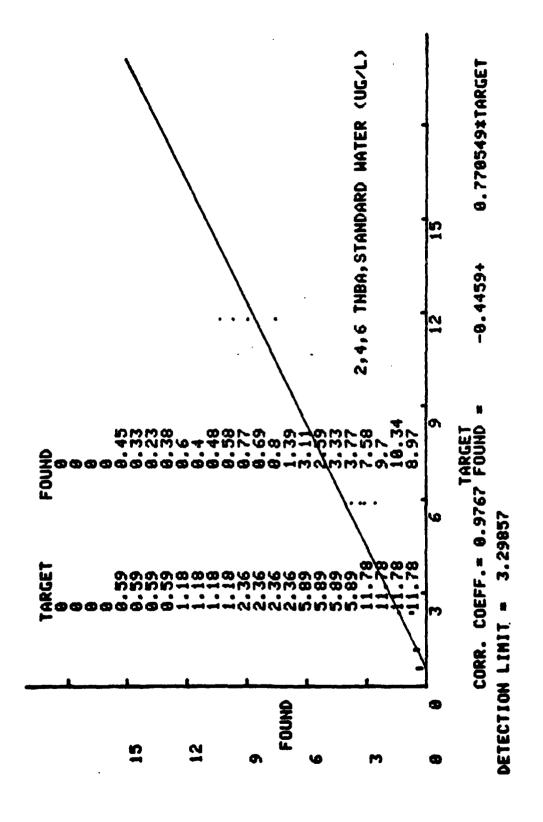


2,4,6 TNBA, NATURAL WATER (UG/L)

Ç,

TARGET CONCENTRATION	1	DAY 2	3	4	10000000
0.000	0.0000	0.0000	0.0000	0.0000	
0.590	0.250	0.290	0.430	0.380	
1.16	0.370	0.470	0.480	0.720	
2.36	1.15	1.13	1.50	1.08	
5.89	4.49	4.95	4.97	4.12	
11.8	9.07	9.96	11.7	9.49	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.590	0.337	0.0822	24.4	-42.7966
1.18	0.510	0.149	29.1	-56.7797
2.36	1.21	0.192	15.8	-48.5170
5.89	4.63	0.407	8.79	-21.3497
11.8	10.0	1.14	11.3	-14.7284



2,4,6 TNBA,STANDARD WATER (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0068	0.0000	0.0000	0.0000	0.0000	
0.590	0.450	0.330	0.230	0.380	
1.18	0.600	0.400	0.480	0.580	
2.36	0.770	0.690	0.800	1.39	
5.89	3.11	2.59	3.33	3.77	
11.8	7.58	9.70	10.3	8.97	

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.000	0.0000	0.0000
0.590	0.347	0.0925	26.6	-41.1017
1.18	0.515	0.0929	18.0	-56.3559
2.36	0.912	0.322	35.3	-61.3348
5.89	3.20	0.491	15.3	-45.6706
11.8	9•15	1.19	13.0	-22.3472

ATNBA IN SOIL SAMPLES

ATNBA IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for ATNBA.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is listed below in ug/g:

 Analyte
 Range (ug/g)

 ATNBA
 0.51 to 10.2

B. SENSITIVITY

The normalized response (integrator counts) at the natural soil detection limit designated in Section IC is listed below:

Analyte Integrator Counts Nanograms
ATNBA 750,970 1,780

The normalized response (integrator counts) at the standard soil detection limit designated in Section 1C is listed below:

 Analyte
 Integrator Counts
 Nanograms

 ATNBA
 429,880
 1,010

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is listed below:

Analyte Detection Limit (ug/g)
ATNBA 3.5

The detection limit in standard soil, calculated according to Hubaux and Vos (1970), is listed below:

Analyte

Detection Limit (ug/g)

ATNBA

2.0

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb light at 254 nm and are extractable from soil with methylene chloride. Interferences are minimized by Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 8 extracts in an 8-hour day. One analyst can perform approximately 8 extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCIATURE AND CHEMICAL ABSTRACT SERVICE (CAS)
REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
ATNBA	2,4,6-trinitrobenzene carbonal	606-34-8
	2,4,6-trinitrobenzene carbox-	
	a 1 dehy de	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

		Melting	Boiling	Density
Analyte	Formula	Point (°C)	Point (°C)	(g/ml)
ATNBA	C7 H2O7N2	119		

Chemical Structure

C. CHEMICAL REACTIONS

ATNBA is explosive and rapidly decomposed by light, and its solutions must be protected by use of amber glass or aluminum foil wrapping. It decomposes rapidly to 135TNB in basic solution via a red-colored intermediate. ATNBA is slowly decomposed to 135TNB by dissolved oxygen, water, or upon heating with water or alcohol.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with an Altex Model 153 UV detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Altex Model 153 UV detector (> = 254 nm)

2. Column: Ultrasphere C18 (4.6-mm ID x 25 cm)

Particle size: 5 um

3. Flow Rate/Mobile Phase: 1 ml/min

45% H₂O/55% methanol

4. Temperature: 25°C

5. Injection Volume: 250 ul, fixed loop

6. Retention Times:

Analyte Retention Time (minutes)
ATNBA 5.8
135TNB (decomposition product) 6.4

C. HARDWARE/GLASSWARE

- 1. 50-ml centrifuge tubes with Teflon@-lined screw caps (8);
- 2. 250-ml boiling flasks (8);
- 3. Glass filter funnels (8);
- 4. Rotary evaporator (8), with controlled water bath, manifold, cold trap, and vacuum pump;
- 5. 15-ml graduated centrifuge tubes (8);
- 6. 5-ml glass syringes with Luer-lock attachments (8); and
- 7. Centrifuge (capacity for 50-ml tubes).

D. CHEMICALS

- 1. Nanograde methylene chloride;
- 2. HPLC-grade methyl alcohol--J.T. Baker Company;
- 3. HPLC-grade water--J.T. Baker Company;
- 4. Reagent-grade anhydrous sodium sulfate;
- 5. Nanograde pentane;
- 6. HPLC-grade acetonitrile--J.T. Baker Company; and
- 7. Florisil® Sep-Paks®--Waters Associates.

4. STANDARDS

A. CALIBRATION STANDARDS

1. The ATNBA stock calib.ation standard (1.02 mg/ml) is prepared by weighing 10.2 mg of ATNBA into a 10-ml

volumetric flask, dissolving the ATNBA in a few ml of acetonitrile, and diluting to the mark with acetonitrile. The stock solution should be stored under nitrogen, in an amber vial, in a freezer. The stock solution should be replaced on a monthly basis or sooner if degradation is observed (bright pink color or appearance of 135TNB on chromatogram).

- 2. An intermediate calibration standard is prepared by diluting 2 ml of the stock solution to 10 ml with acetonitrile. This standard should be stored under nitrogen, in a septumsealed, amber vial in the freezer. The standard should be replaced on a weekly basis or sooner if degradation is observed.
- 3. Prepare a series of working calibration standards by making dilutions of the intermediate calibration standard. The working calibration standards should be made fresh just prior to analysis. Dilute with a 50% methanol/50% water solution which has been vacuum degassed and stored under nitrogen. Prepare the dilutions as follows:

Working Calibration Standard	Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
A	Intermediate Calibration Standard	1	10
В	Intermediate Calibration Standard	0.5	10
С	Intermediate Calibration Standard	0.1	5
מ	Intermediate Calibration Standard	0.1	10
E	Intermediate Calibration Standard	0.05	10
P	Working Calibration Standard C	1	10

Working Calibration Standard	Concentration (ug/ml)	
A	20.4	
В	10.2	
С	4.08	
ם	2.04	
E	1.02	
F	0.408	

B. CONTROL SPIKES

- 1. Use the intermediate calibration standard as the control spiking solution.
- Allow the soil sample to air dry on the dull side of aluminum foil until it can be sieved through a 30-mesh sieve. (Sediment samples are extracted wet.)
- 3. Weigh 20 g of sieved soil or wet sediment into a 50-ml centrifuge tube with a Teflon®-lined screw cap.
- 4. Pipette a known amount of the control spike solutions into 3 ml of methylene chloride and quantitatively transfer to the 20-g soil sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

Spike Volume (ml)	Concentration of Spiked Soil (ug/g)			
0.05	0.51			
0.1	1.02			
0.2	2.04			
0.5	5.1			
1.0	10.2			

- 5. Allow the soil to air dry for at least 1 hour. Shake the soil to ensure mixing of the spiking solution throughout the sample.
- 6. Extract the sample according to the procedure presented in Section 5B.

5. PROCEDURE

A. SAMPLE PREPARATION

- 1. Let all soil samples air dry on the dull side of aluminum foil until they are suitably dry so they can be sieved through a 30-mesh sieve.
- 2. Sample the sieved soil by quartering and weighing 20 g into a 50-ml centrifuge tube.

B. EXTRACTION

- 1. Add 30 ml of methylene chloride to the centrifuge tube.
- Cap the tube, shake for 3 minutes, and centrifuge for 5 minutes at 2,000 rpm.
- 3. Decant off the methylene chloride and pass through a glass funnel filled with a small plug of glass wool and approximately 20 g of anhydrous sodium sulfate into a 250-ml boiling flask. In event of a wet sediment, the methylene chloride layer should be withdrawn by use of a Pasteur pipet and the aqueous layer retained.
- 4. Steps 1 through 3 should be repeated twice more and the methylene chloride extracts combined. The extracts in the boiling flask should be protected from light by covering with aluminum foil.
- 5. After the third extract has been transferred to the boiling flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
- 6. Concentrate the methylene chloride extract by rotary evaporation on a 35°C water bath.
- 7. When the apparent volume of the solution remaining in the flask is about 1 ml, remove the apparatus from the rotovap. Transfer the extract to a 15-ml graduated centrifuge tube using a Pasteur pipet. Rinse the boiling flask with an additional 1 ml of methylene chloride, and transfer the rinses to the centrifuge tube. Raise the volume of the extract to 2.5 ml with methylene chloride.

7

- Raise to a final volume of 5 ml by adding nanograde pentane.
 Mix.
- 9. Equip a 5-ml glass syringe with a Florisil® Sep-Pak®. Pour the extract into the syringe and pass through the Sep-Pak®, discarding the eluent.
- 10. Put 5 ml of a 20% acetonitrile/80% methylene chloride solution in the centrifuge tube and pour into the syringe. Elute the Sep-Pak® into the original boiling flask.
- 11. Evaporate the extract on a rotovap to an approximate volume of 1 ml. Add 10 ml of acetonitrile, and reduce the volume to 1 ml. Repeat with a further addition of 10 ml of acetonitrile and a final volume of 1 ml.
- 12. Transfer the 1-ml extract to a 15-ml graduated centrifuge tube using a Pasteur pipet. Rinse the flask with an additional 2 ml of acetonitrile (which has been vacuum degassed and stored under nitrogen) and transfer to the centrifuge tube. Raise to a volume of 4 ml with acetonitrile and to a final volume of 10 ml with HPLC-grade water (vacuum degassed and stored under nitrogen).
- 13. Transfer to a 5-m1 amber, septum-sealed vial for storage at 4°C. Fill the vial to minimize headspace.
- 14. The extract is now ready for chromatography by HPLC. The extract should be analyzed within 24 hours and preferably immediately.

C. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC coluan.
- 2. Perform the analysis of the sample using the conditions given in Section 3B.
- 3. Measure the response of the component of interest.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of ATNBA according to the following formula:

Concentration (ug/g) =
$$\frac{(A)(V_t)}{W_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve,

 $V_t = Volume of total extract (ml), and$

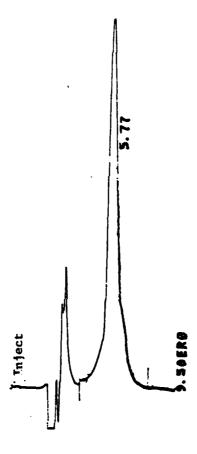
 W_s = Weight of initial sample extracted (g).

7. REFERENCES

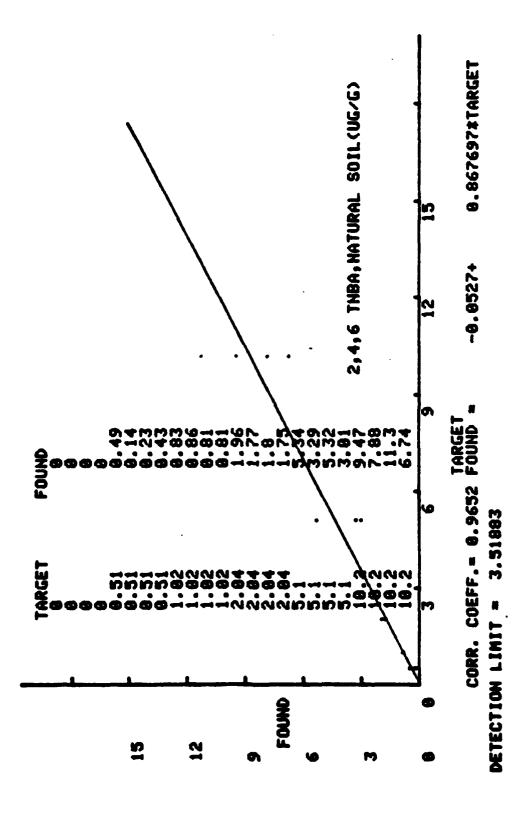
None found.

8. DATA

See attached data sheets.



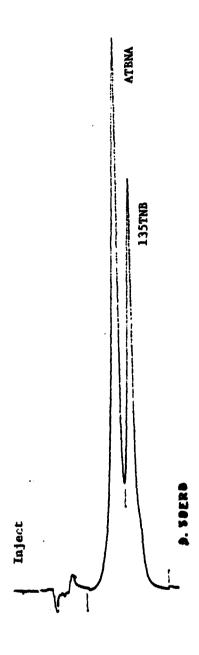
Chromatogram of an Extract of Natural Soil Spiked with 2.04 ug/g of ATNBA



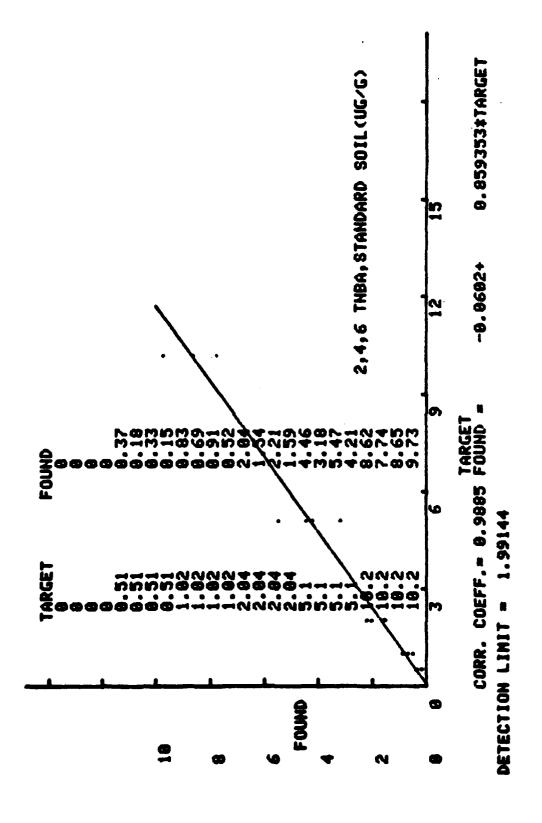
2.4.6 Theathatural soll(UG/G)

Thedat Curcenthation	1	2 DAY	5	4	
1.0051	9.3500	3.3000	0.000 J	0.0016	
0.510	0.490	0.140	0.230	453	
1.02	3.838	0.860	0.816	3.610	
2 • 2 4	1.96	1.77	1.80	1.75	
5.10	5.34	3.29	5.32	3.01	
13.2	5.47	7.88	11.3	6.74	

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT Inaccuracy
0.0000	0.0000	0.0000	0.0000	0.0000
0. 51 0	9 .32 2	5.165	51.1	-36.7647
1.62	2.827	0.0236	2.85	-18.8726
2.04	1.82	0.0956	5.25	-10.7843
5.10	4.24	1.26	29∙8	-16.6627
13.2	8.85	1.98	22.4	-13.2598



Chromatogram of ATNBA and 135TNB (Decomposition Product) under the Conditions Given in Section 3B



2:4.6 TYPA.STANDARD SCIL(UG/G)

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TIPDET CUP CENTRATION	1	DAY 2	3	4	
0.0130	0.0000	0.0000	0.0000	0.9500	
0.810	0.370	0.180	0.330	0.150	
1.72	0.830	0.690	0.910	0.520	
2.04	2.04	1.54	2.21	1.59	
5 - 10	4.46	3.18	5.47	4.21	
1:•2	8.62	7.74	8.65	9.73	

TAPGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	-
0.0000	S.000G	0.0000	0.0000	0.0000	
.51.	€.257	0.109	42.2	-49.5098	
1 • 5 2	:•737	0.171	23.2	-27.6561	
2.04	1.84	0.331	18.0	-9.5588	
5.15	4.33	0.946	21.7	-15.0980	
1:.2	8 • 68	0.815	9.38	-14.8529	

35DNP IN WATER SAMPLES

35DNP IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for 35DNP.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is 0.61 to 12.2 ug/L.

B. SENSITIVITY

The normalized response (integrator counts) at the natural water detection limit designated in Section 1(C) is listed below:

Analyte	Integrator Counts	Nanograms
35DNP	69,000	510

The normalized response (integrator counts) at the standard water detection limit designated in Section 1(C) is listed below:

Analyte	Integrator Counts	Nanograms	
35DNP	58,000	431	

C. DETECTION LIMIT

The detection limit in natural water calculated according to Hubaux and Vos (1970), is 4.5 ug/L. The detection limit in standard water is 3.8 ug/L.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile, water soluble organic compounds which absorb light at 340 nm and are extractable from water with methylene chloride. Interferences are minimized by a base-neutral pre-extraction of the sample and a Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 extracts in an 8-hour day. One analyst can perform approximately six extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCIATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
35DNP	None	586-11-8

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

			Acid
		Melting	Dissociation
Analyte	Formula	Point (°C)	Constant
35DNP	C6 H305 N2	126	pk = 6.7

Chemical Structure

C. CHEMICAL REACTIONS

35DMP undergoes normal phenolic reactions such as acid dissociation. No reactions which adversely affected the stability of the compound were observed.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual pump liquid chromatograph equipped with an Altex Model 153 UV detector and 340-mm filter interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

- 1. Detector: Altex Model 153 fixed-wavelength detector equipped with a 340-nm filter. (λ = 340 nm)
- 2. Column: Ultrasphere C18 (4.6-mm ID x 25 cm)

Particle size: 5 um

3. Flow Rate/Mobile Phase: | ml/min

30% water/70% methanol/

0.03 M H3PO4

- 4. Temperature: 25°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Time: 6.0 minutes

C. HARDWARE/GLASSWARE

- 1. 1-L Teflon separatory funnels with screw caps (6);
- 2. Short-stemmed glass funnels (6);
- 3. 500-ml K-D evaporative flasks, acid washed (6);
- 4. 3-ball Snyder columns (6);
- 5. 2-ball micro-Snyder columns (v);
- 6. 10-ml graduated centrifuge tubes (6);
- 7. 5-ml glass syringes with Luer-lock tips (6); and
- 8. 1-liter graduated cylinders (6).

D. CHEMICALS

- 1. Nanograde methylene chloride;
- 2. HPLC-grade methyl alcohol--J.T. Baker Company;
- 3. HPLC-grade water--J.T. Baker Company;
- 4. Acid-washed, anhydrous sodium sulfate (reagent grade)—
 prepared as follows: in a 500-ml, round-bottom flask,
 slurry 100 g of anhydrous sodium sulfate with 200 ml of
 diethyl ether containing 0.1 ml of concentrated sulfuric
 acid. Attach the flask to a rotary evaporator and remove
 the ether by vacuum evaporation. Store the treated sodium
 sulfate at 130°C.

- 5. Acid-treated glass wool--Supelco 2-0383;
- 6. 6N NaOH--weigh out 240 g of reagent-grade NaOH pellets and dissolve in 1 L of organic-free water;
- 7. 85% phosphoric acid, reagent grade;
- 8. 6N HCl--dilute concentrated HCl 1:1 with water;
- 9. Florisil[●] Sep-Paks[®]--Waters Associates;
- 10. Sodium chloride, reagent grade;
- 11. Teflon® boiling chips; and
- 12. ColorpHast® pH indicator sticks--MC/B Manufacturing Chemists, Inc.

4. STANDARDS

A. CALIBRATION STANDARDS

- The stock calibration standard (1.22 mg/ml) is prepared by weighing 12.2 mg of 35DNP into a 10-ml volumetric flask, dissolving in a few ml of methanol, and diluting with methanol to the mark.
- 2. Prepare a series of working calibration standards by making dilutions of the stock calibration standard. Dilute with the HPLC mobile phase (30% water/70% methanol/0.03 M H₃PO₆) as follows:

Working Calibration Standard	Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
A	Stock Calibration Standard	1	50
В	Standard A	5	10
C	Standard A	1	10
D	Standard A	2	25
E	Standard A	1	25
*	Standard A	1	50

Working Calibration Standard	Concentration (ug/ml)
A	24.4
В	12.2
С	2.44
D	1.95
E	0.976
F	0.488

B. CONTROL SPIKES

- 1. Use Working Calibration Standard A as the control spike solution.
- Measure 900 ml of water into a 1-L Teflon[●] separatory funnel.
- 3. Pipet a known amount of the control spike solution into the 900-ml sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

Spike Volume (m1)	Concentration of Spiked Water (ug/L)		
0.05	1.36		
0.10	2.71		
0.20	5.42		
0.50	13.6		
1.00	27.1		

4. Shake the sample to assure a homogeneous mixture before extraction.

5. PROCEDURE

A. EXTRACTION

- Measure 900 ml of sample to be analyzed and pour into a 1-L separatory funnel.
- Adjust the sample pH to 12 with 6N NaOH (approximately 3 ml). Add 100 g of NaCl to the sample. Shake the sample to dissolve the salt.

- 3. Add 100 ml of methylene chloride and shake for 1 minute. Allow the layers to separate at least 10 minutes. Centrifugation or placing the separatory funnel in an ultrasonic bath aids in breaking any emulsions.
- 4. Discard the methylene chloride extract.
- 5. Add 3 ml of 85% H₃PO₄ to the sample. Mix well and then adjust the pH to 3 by adding 6N NaOH (approximately 3.5 ml).
- Extract the sample sequentially with three 100-ml portions of methylene chloride using 2-minute shake times and 10-minute separation times.
- 7. Pass the extracts through a glass funnel containing approximately 20 g of acid-washed sodium sulfate and an acid-washed glass wool plug. Collect the extracts in an acid-washed K-D apparatus with a 10-ml receiver. Rinse the sodium sulfate with approximately 20 ml of methylene chloride. Add a Teflon® boiling chip to the extract, and attach a three-ball Snyder column to the apparatus.
- 8. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath, immersing the receiver nearly up to the joint. The balls in the Snyder column should chatter actively at the proper evaporation rate.
- 9. Let the apparent volume of extract in the receiver decrease to approximately 2 ml, then remove the receiver from the bath and let cool. Detach the receiver from the K-D apparatus and add methylene chloride to raise the volume to approximately 5 ml.
- 10. Attach a Florisil® Sep-Pak® to a 5-ml syringe and transfer the extract into the syringe. Pass the entire extract through the Sep-Pak® at a rate of approximately 5 ml/min and discard the eluate. Rinse the receiver with an additional 1 ml of methylene chloride, pass the rinses through the Sep-Pak®, and discard the eluate.

- 11. Elute the analyte from the Sep-Pak® with 5 ml of 5% methanol in methylene chloride and collect in the original receiver.
- 12. Add approximately 2 ml of methanol and a fresh Teflon® boiling chip to the receiver and attach the two-ball micro-Snyder column. Concentrate to approximately 2 ml on a 90°C water bath. Repeat the concentration by adding 10 ml of methanol and reconcentrating to approximately 2 ml. Repeat this process once more by adding 10 ml of methanol and reducing the volume to less than 1.0 ml.
- 13. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing the receiver with 0.5 ml of HPLC-grade water. Add two drops of 85% phosphoric acid and raise the extract volume to exactly 2.0 ml in the centrifuge tube with HPLC-grade water.
- 14. Transfer to a 2-ml septum-sealed vial for storage at 4°C.

 The extract is now ready for HPLC analysis.

B. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample using the conditions given in Section 3(B).
- 3. Measure the response of the 35DNP peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of 35DNP according to the following formula:

Concentration (ug/L) =
$$\frac{(A)(V_t)}{V_s}$$

 V_t = Volume of total extract (m1), and

 V_s * Volume of initial sample extracted (L).

7. REFERENCES

None found.

8. DATA

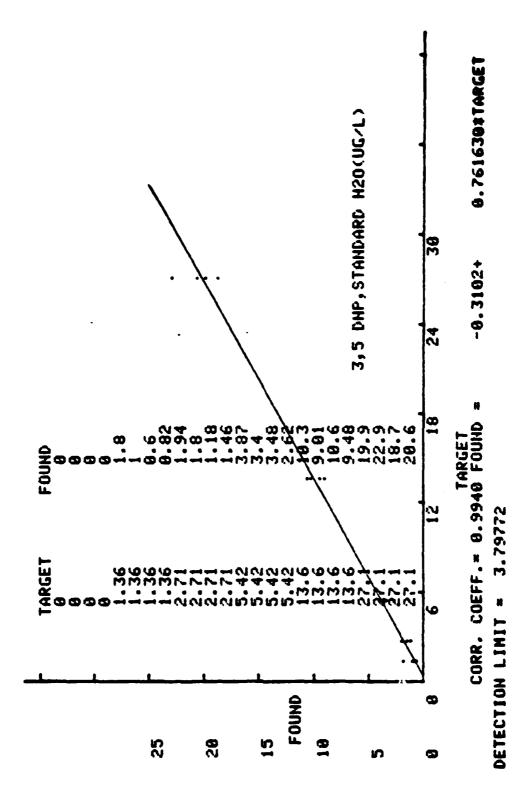
See attached data sheets.

3.5 FOR .STANDARD F20(UG/L)

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TARGET CONCENTRATION	1	DAY 2		4	
2.7111	0.0001	1.9930	9.5000	0.9336	
1.35	1.90	1.000	0.600	0.823	
2.71	1.94	1.09	1.16	1.46	
5.42	3.87	3.40	3.48	2.62	
13-5	10.3	9.01	10.6	9.48	
27.1	19.9	22.9	18.7	20.6	

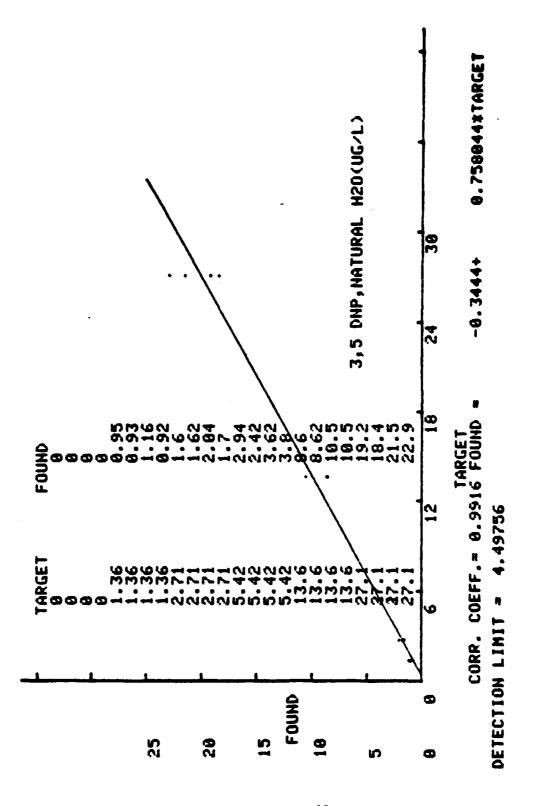
TARGET CONCENTS ATIO	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
1.0000	5.906	3 • 2 2 2 9	0.0000	0.0000
1.00	1.15	0.523	49•6	-22.4265
2.7.	1.55	0.342	21.5	-41-1439
5 . 4 €	3.34	9.524	15.7	-38.3363
13.6	9.35	ċ• 7 32	7.43	-27.5919
27.1	20.5	1.77	6.61	-24.2629



3,5 DMP, MATURAL H20(UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.000	0.0000	0.0000	0.0000	0.0000	
1.36	0.950	0.930	1.16	0.920	
2.71	1.60	1.62	2.04	1.70	
5.42	2.94	2.42	3.62	3.80	
13.6	8.60	8.62	10.5	10.5	
27.1	19.2	18.4	21.5	22.9	

TARGET CONCENTRATION	AVERAGE Found value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.3000	0.000	0.0000	0.000
1.36	3.990	0.114	11.5	-27.2059
2.71	1.74	0.205	11.8	-35.7934
5.42	3.19	0.636	19.9	-41.0517
13.6	9.55	1.09	11.4	-29.7426
27.1	20.5	2.07	10.1	-24.3543



35DNP IN SOIL SAMPLES

35DNP IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for 35DNP.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is 0.61 to 12.2 ug/g.

B. SENSITIVITY

The normalized response (integrator counts) at the natural soil detection limit designated in Section 1(C) is listed below:

Analyte	Integrator Counts	Nanograms
35DNP	125,596	850

The normalized response (integrator counts) at the standard soil detection limit designated in Section 1(C) is listed below:

Analyte	Integrator Counts	Nanograms
35DNP	140,160	950

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is 1.7 ug/g. The detection limit in standard soil is 1.9 ug/g.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile, water soluble organic compounds which absorb light at 340 nm and are extractable from water with methylene chloride. Interferences are minimized by a water extraction of the sample and a Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 extracts in an 8-hour day. One and out can perform approximately six extractions in an 8-moder day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS)
REGISTRY NUMBER

		CAS REGISTRY
Analyte	Alternate Nomenclature	Number
35DNP	None	586-11-8

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

Analyte	Formula	Melting Point (°C)	Acid Dissociation Constant	
35DNP	C6 H305N2	126	pk _a = 6.7	

Chemical Structure

C. CHEMICAL REACTIONS

35DMP undergoes normal phenolic reactions such as acid dissociation. No reactions which adversely affected the stability of the compound were observed.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with an Altex Model 153 UV detector and 340-nm filter interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

- 1. Detector: Altex Model 153 fixed-wavelength detector equipped with a 340-nm filter (λ = 340 nm)
- 2. Column: Ultrasphere C18 (4.6-mm ID x 25 cm)

Particle size: 5 um

3. Flow Rate/Mobile Phase: 1 ml/min

30% water/70% methanol

0.03 M H3PO4

- 4. Temperature: 25°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Time: 6.0 minutes

C. HARDWARE/GLASSWARE

- 1. 250-ml separatory funnels with stoppers, acid washed (6);
- 2. Short-stemmed glass funnels (6);
- 3. 500-ml K-D evaporative flasks, acid washed (6);
- 4. 3-ball Sayder columns (6);
- 5. 2-ball micro-Snyder columns (6);
- 10-ml graduated centrifuge tubes (6);
- 7. 5-ml glass syringes with Luer-lock tips (6);
- 8. I-L graduated cylinders (6); and
- 9. 50-ml centrifuge tubes with Teflon -lined screw caps (6).

D. CHEMICALS

- 1. Nanograde methylene chloride;
- 2. HPLC-grade methyl alcohol--J.T. Baker Company;
- 3. HPLC-grade water--J.T. Baker Company;
- 4. Acid-washed, anhydrous sodium sulfate (reagent grade)—
 prepared as follows: in a 500-ml, round-bottom flask,
 slurry 100 g of anhydrous sodium sulfate with 200 ml of
 diethyl ether containing 0.1 ml of concentrated sulfuric
 acid. Attach the flask to a rotary evaporator and remove
 the ether by vacuum evaporation. Store the treated sodium
 sulfate at 130°C.

- 5. Acid-trea i glass wool--Supelco 2-0383;
- 6. 85% phosphoric acid, reagent grade;
- 7. 6N HCl-- ite concentrated HCl 1:1 with water for acid washing gl ware;
- 8. Florisil Sep-Paks -- Waters Associates;
- 9. Sodium chloride, reagent grade;
- 10. Teflon boiling chips; and
- 11. ColorpHast® pH indicator sticks--MC/B Manufacturing Chemists, Inc.

4. STANDARDS

A. CALIBRATION STANDARDS

- The stock calibration standard (1.22 mg/ml) is prepared by weighing 12.2 mg of 35DNP into a 10-ml volumetric flask, dissolving in a few ml of methanol, and diluting with methanol to the mark.
- 2. Prepare a series of working calibration standards by making dilutions of the stock calibration standard. Dilute with the HPLC mobile phase (30% water/70% methanol/0.03 M H₃PO₄) as follows:

Working Calibration Standard Standard Diluted		Volume of Standard Used (ml)	Final Volume (ml)	
A	Stock Calibration Standard	5	25	
В	Standard A	1	10	
C	Standard A	0.5	10	
D	Standard B	2	10	
E	Standard B	1	10	
F	Standard B	0.5	10	
G	Standard C	0.5	10	

Working Calibration Standard	Concentration (ug/ml)
A	244
В	24.4
С	12.2
מ	4.88
E	2.44
F	1.22
G	0.61

B. CONTROL SPIKES

- 1. Use Working Calibration Standard A as the control spike solution.
- 2. Weigh out 20.0 g of soil into a 50-ml centrifuge tube.
- 3. Pipet a known amount of the control spike solution (dissolved in a sufficient volume of HPLC-grade water to just wet the soil) into the centrifuge tube. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

Volume of Control Spike Solution (ml)	Concentration of Spiked Soil (ug/L)		
0.05	0.61		
0.10	1.22		
0.20	2.44		
0.50	6.1		
1.00	12.2		

4. Shake the sample to mix and let set I hour to air dry.

5. PROCEDURE

A. SAMPLE PREPARATION

1. Let soil sample air dry on dull side of aluminum foil until it can be sieved through a 30-mesh sieve.

2. Sample the sieved soil by quartering and place 20.0 g into a 50-ml centrifuge tube.

B. EXTRACTION

- 1. Add 35 ml of HPLC-grade water to the centrifuge tube and mix thoroughly by shaking for 3 minutes. Centrifuge the sample at approximately 3,000 rpm for 15 minutes, and decant the water layer into a 250-ml separatory funnel.
- 2. Repeat Step 1 twice more and combine the extracts.
- 3. Adjust the pH of the water extracts to 3 with 85% phosphoric acid.
- 4. Extract the water extracts sequentially with three 80-ml portions of methylene chloride using 2-minute shake times and 10-minute separation times.
- 5. Pass the methylene chloride extracts through a glass funnel containing approximately 20 g of acid-washed sodium sulfate and an acid-washed glass wool plug. Collect the extracts in an acid-washed K-D apparatus with a 10-ml receiver. Rinse the sodium sulfate with approximately 20 ml of methylene chloride. Add a Teflon® boiling chip to the extract, and attach a three-ball Snyder column to the apparatus.
- 6. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath, immersing the receiver nearly up to the joint. The balls in the Snyder column should chatter actively at the proper evaporation rate.
- 7. Let the apparent volume of extract in the receiver decrease to approximately 2 ml, then remove the receiver from the bath and let cool. Detach the receiver from the K-D apparatus and add methylene chloride to raise the volume to approximately 5 ml.
- 8. Attach a Florisil® Sep-Pak® to a 5-ml syringe and transfer the extract into the syringe. Pass the entire extract through the Sep-Pak® at a rate of approximately 5 ml/min and discard the eluate. Rinse the receiver with an additional

- l ml of methylene chloride, pass the rinses through the Sep-Pak®, and discard the eluate.
- 9. Elute the analyte from the Sep-Pak® with 5 ml of 5% methanol in methylene chloride and collect in the original receiver.
- 10. Add approximately 2 ml of methanol and a fresh Teflon[®] boiling chip to the receiver and attach the two-ball micro-Snyder column. Concentrate to approximately 2 ml on a 90°C water bath. Repeat the concentration by adding 10 ml of methanol and reconcentrating to approximately 2 ml. Repeat this process once more by adding 10 ml of methanol and reducing the volume to less than 2.0 ml.
- 11. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing the receiver with 2-ml of HPLC-grade water. Add two drops of 85% phosphoric acid and raise the extract volume to exactly 10.0 ml in the centrifuge tube with HPLC-grade water.
- 12. Transfer a portion of the sample to a 2-ml septum-sealed vial for storage at 4°C. The extract is now ready for HPLC analysis.

B. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample using the conditions given in Section 3(B).
- 3. Measure the response of the 35DNP peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of 35DNP according to the following formula:

Concentration (ug/g) =
$$\frac{(A)(V_t)}{(W_g)}$$

 V_t = Volume of total extract (m1), and

Ws = Weight of initial sample extracted (g).

7. REFERENCES

None found.

8. DATA

See attached data sheets.

Chromatogram of an Extract of a Natural Soil Sample Spiked with 35DNP at 1.2 ug/g

3.5 CINITROPHENOL, STD. SOIL (UG/G)

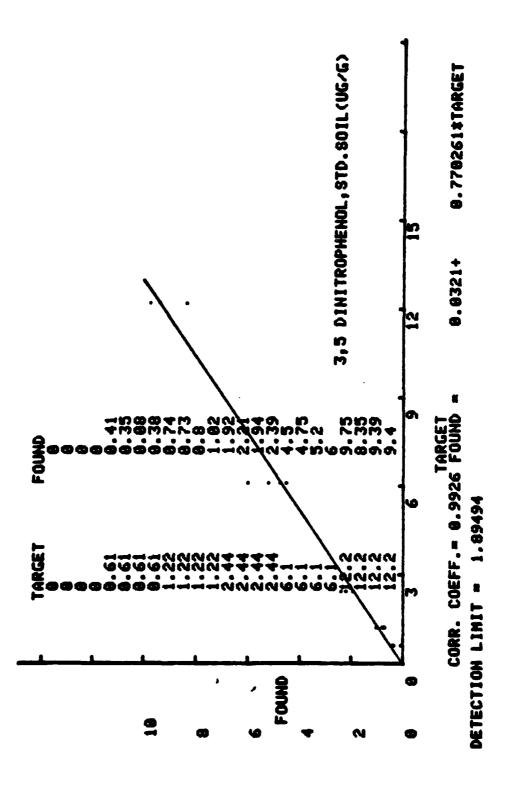
TARGET CONCENTRATION	1	DAY 2	3	4	
0.000	0.0000	0.0000	0.0000	0.0000	
0.610	0.410	0.350	0.0800	0.380	
1.22	0.746	0.730	0.800	1.02	
2.44	1 • 92	2.21	1.94	2.39	
6.10	4.50	4.75	5.20	6.00	
12.2	9.75	8.35	9.39	9.40	

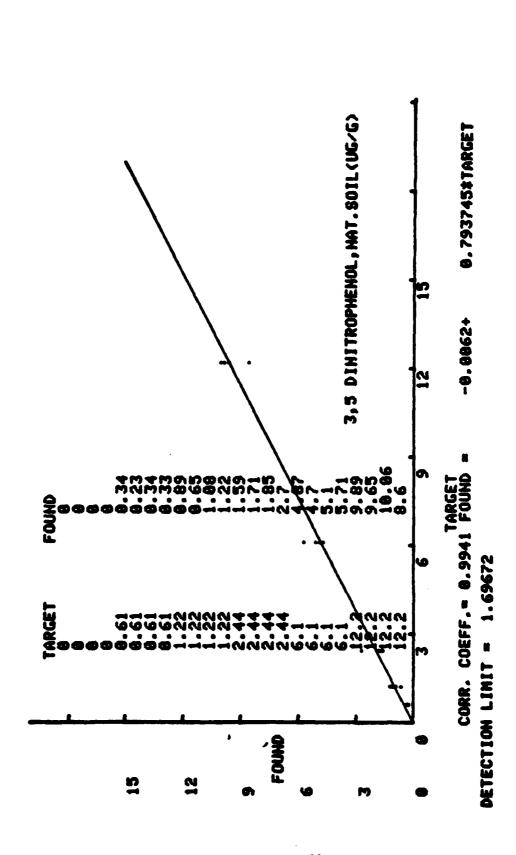
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.020.0	0.0000	0.000	0.0000	0.0000
0.610	0.305	0.152	49.8	-50.0000
1.22	0 -822	0.135	16.4	-32.5820
2.44	2.11	0.226	10.7	-13.3197
6.13	5.11	0.659	12.9	-16.1885
12.2	9.22	0.605	6.56	-24.4057

3.5 DINITROPHENCL.NAT.SOIL(UG/G)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	0.0000	
0.610	0.340	0.230	0.340	0.330	
1.22	0.890	0.650	1.08	1.22	
2.44	1.59	1.71	1.85	2.70	
6.10	4.87	4.70	5.10	5.71	
12.2	9.89	9 • 65	10.1	8.60	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0.0000	0.0000	0.0000	0.000	
0.610	0.310	0.0535	17.3	-49.1803	
1.22	0.960	0.247	25.7	-21.3115	
2.44	1.96	0.503	25•6	-19.5697	
6.10	5.09	0.442	8.67	-16.4754	1
12.2	9.55	0.655	6.86	-21.7213	





35DNA IN WATER SAMPLES

35DNA IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for 35DNA.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard water is 0.58 to 11.7 ug/L.

B. SENSITIVITY

The normalized response (integrator counts) at the natural water detection limit designated in Section 1(C) is listed below:

Analyte	Integrator Counts	Nanograms
35DNA	89,900	342

The normalized response (integrator counts) at the standard water detection limit designated in Section 1(C) is listed below:

Analyte	Integrator Counts	Nanograms
35DNA	31,300	120

C. DETECTION LIMIT

The detection limit in natural water calculated according to Hubaux and Vos (1970), is 3.0 ug/L. The detection limit in standard water is 1.1 ug/L.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile basic and neutral organic compounds which absorb light at 395 nm and are extractable from water with methylene chloride. Interferences are minimized by Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

2. CHEMISTRY

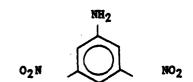
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

Analyte	Alternate Nomenclature	Number
35DNA	3,5-dinitroaniline,	618-87-1
	1-amino-3,5-dinitrobenzene	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

			Acid
Analyte	Formula	Melting Point (°C)	Dissociation Constant
35DNA	C6 H5 O4 N3	160-162	$pk_a = 0.23$

Chemical Structure



C. CHEMICAL REACTIONS

35DNA reacts as a weak base.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

- 1. Detector: Perkin-Elmer LC-75 variable-wavelength detector $(\lambda = 395 \text{ nm})$
- 2. Column: Ultrasphere ODS (4.6-mm ID x 25 cm

Particle size: 5 um

3. Flow Rate/Mobile Phase: 1 ml/min

30% water/70% methanol

- 4. Temperature: 25°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Time: 5.6 minutes

C. HARDWARE/GLASSWARE

- 1. 1-L glass separatory funnels with stoppers (8);
- 2. Short-stemmed glass funnels (8);
- 3. 500-ml K-D evaporative flasks (8);
- 4. 25-ml graduated K-D receivers (8);
- 5. 3-ball Snyder columns (8);
- 6. 2-ball micro-Snyder columns (8);
- 7. 10-ml graduated centrifuge tubes (8);
- 8. 5-ml glass syringes with Luer-lock attachments(8);
- 9. 1-L graduated cylinders (8); and
- 10. 100-ml graduated cylinder (1).

D. CHEMICALS

- 1. Nanograde methylene chloride;
- 2. HPLC-grade methyl alcohol--J.T. Baker Company;
- 3. HPLC-grade water--J.T. Baker Company;
- 4. Anhydrous sodium sulfate, reagent grade;
- 5. Glass wool;
- Teflon[●] boiling chips;
- 7. Florisil® Sep-Paks®--Waters Associates;

- 8. 6N sodium hydroxide--weigh out 240 g of reagent-grade NaOH pellets and dissolve in 1 L of HPLC-grade water; and
- 9. ColorpHast[●] pH indicator sticks--MC/B Manufacturing Chemists, Inc.

4. STANDARDS

A. CALIBRATION STANDARDS

- The 35DNA stock calibration standard (1.05 mg/ml) is prepared by weighing 10.5 mg of 35DNA into a 10-ml volumetric flask, dissolving the 35DNA in a few ml of methanol, and diluting with methanol to the mark.
- 2. Prepare a series of working calibration standards by making dilutions of the stock calibration standard. Dilute with 50% methanol/50% water as follows:

Working Calibration Standard	Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
A	Stock Calibration Standard	1	100
В	Standard A	5	10
C	Standard A	i	10
D	Standard A	2	25
E	Standard A	1	25
P	Standard A	1	50

Working Calibration Standard	Concentration (ug/ml)
A .	10.5
В	5.25
C	1.05
D	0.84
E	0.42
F	0.21

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B. CONTROL SPIKES

- 1. Use Working Calibration Standard A as the control spike solution.
- 2. Measure 900 ml of water into a 1-L separatory funnel.
- 3. Pipet a known amount of the control spike solution into the 900-ml sample. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

Spike Volume (m1)	Concentration of Spiked Water (ug/L)
0.05	0.58
0.10	1.17
0.20	2.33
0.50	5.83
1.00	11.67

4. Shake the sample to assure a homogeneous mixture before extraction.

5. PROCEDURE

A. EXTRACTION

- Measure 900 ml of sample to be analyzed and pour into a l-L separatory funnel.
- 2. Adjust the sample pH to 12 with 6N NaOH (approximately 3 ml).
- 3. Extract the sample sequentially with three 100-ml portions of methylene chloride using 2-minute shake times and 10-minute separation times. Centrifuge or sonicate to separate any resulting emulsions.
- 4. Pass the extracts through a glass funnel containing approximately 20 g of sodium sulfate and a glass wool plug. Collect the extracts in a K-D apparatus equipped with a 10-ml receiver. Rinse the sodium sulfate with approximately

- 20 ml of methylene chloride. Add a Teflon® boiling chip to the extract, and attach a three-ball Snyder column to the apparatus.
- 5. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath, immersing the receiver nearly up to the joint. The balls should chatter actively at the proper evaporation rate.
- 6. Let the apparent volume of extract decrease to approximately 2 ml, then remove from the bath, and let cool. Remove the receiver from the K-D apparatus and add methylene chloride to raise the volume to approximately 5 ml.
- 7. Attach a Florisil® Sep-Pak® to a 5-ml syringe and transfer the extract into the syringe. Pass the entire extract through the Sep-Pak® at a rate of approximately 5 ml/min and discard the eluate. Rinse the receiver with an additional 1 ml of methylens chloride, pass the rinses through the Sep-Pak®, and discard the eluate.
- 8. Elute the analyte from the Sep-Pak® with 5 ml of 5% methanol in methylane chloride and collect in the original receiver.
- 9. Add approximately 2 ml of methanol and a fresh Teflon® boiling chip to the receiver, and attach the two-ball micro-Snyder column. Concentrate to approximately 2 ml on a 90°C water bath. Add an additional 10 ml of methanol and reconcentrate to approximately 2 ml. Repeat this process once more by adding 10 ml of methanol and concentrating to a volume of approximately 0.5 ml.
- 10. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing the receiver quantitatively with 0.5 ml of HPLC-grade water. Raise the extract volume to exactly 2.0 ml in the centrifuge tube with HPLC-grade water.
- 11. Transfer to a 2-ml septum-sealed vial for storage at 4°C.
- 12. The extract is now ready for HPLC analysis.

C. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample using the conditions given in Section 3(B).
- 3. Measure the response of the 35DNA peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of 35DNA according to the following formula:

Concentration (ug/L) =
$$\frac{(A)(V_t)}{V_s}$$

 V_t = Volume of total extract (m1), and

 V_s = Volume of initial sample extracted (L).

7. REFERENCES

None found.

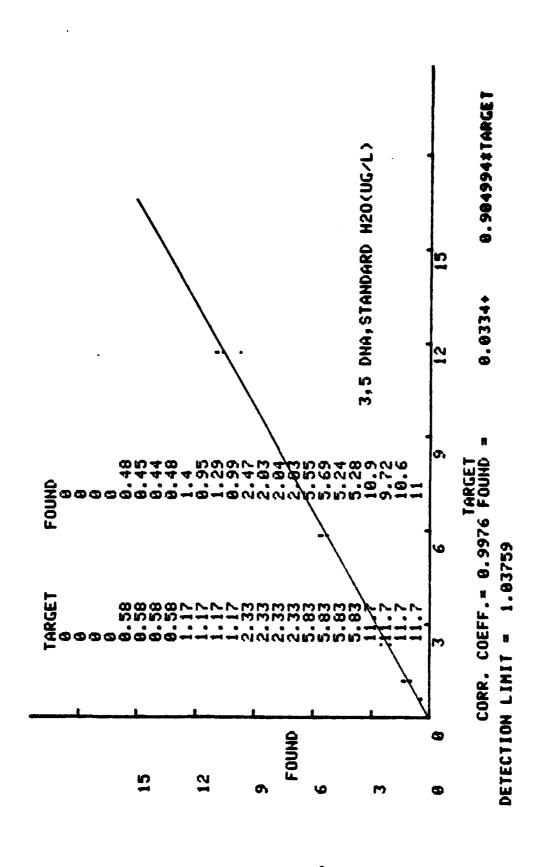
8. DATA

See attached data sheets.

3.5 CMA.STANDAPE H20(UG/L)

TARGET CONCEPTRATION		DAY 2	3	4	
3.0000	0.0000	0.0000	0.0000	0.0000	
0.560	0.480	0.450	0.440	0.480	
1.17	1.40	0.950	1.25	0.996	
2.33	2.47	2.03	2.04	2.03	
5.83	5.55	5.69	5.24	5.28	
11.7	10.9	9.72	10.6	11.0	

TARGET CONCELIPATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
3.1313	5 4 6 6 3 C	0.0000	0.0000	0.0000
^•5.0	.462	0.0206	4.46	-20.2586
1 - 17	1.16	0.222	19.2	-1.0684
2.33	2.14	0.218	10.2	-8.9472
5•€3	5.44	0.216	3.57	-6.6895
11.7	13.6	9.582	5.51	-9. 7863



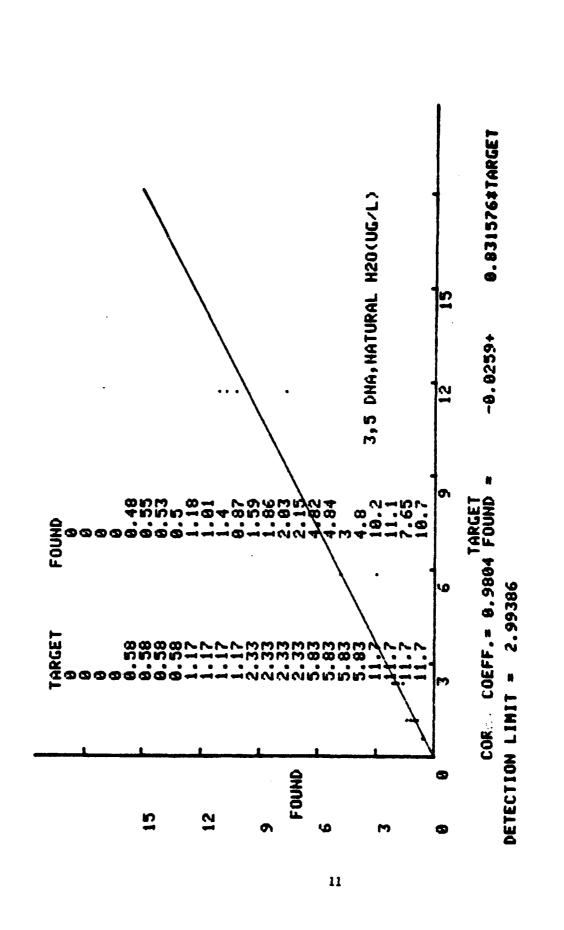
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3.5 CNA.MATUFAL H20(UG/L)

TARGET CONCENTRATION	1	DAY 2		4	
. 0.0000	C.000G	0.0000	0.9620	0.0000	
0.500	0.483	0.550	0.530	0.500	
1.17	1.18	1.01	1.40	0.870	
2.33	1.59	1.86	2.03	2.15	
5.83	4.82	4.84	3.00	4 • 8 C	
11.7	10.2	11.1	7.65	10.7	

TARGET CONCEMTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
5.(300	0.0063	0.0000	0.000	0.0000
5 .5 85	∌ • 515	0.9311	6 • 6 4	-11-2069
1.17	1.11	0.228	20.5	-4.7009
2.33	1.91	0.243	12.7	-18.1331
5.53	4.36	0.510	20.5	-25.1267
11.7	9.91	1.55	15.7	-15.2778



35DNA IN SOIL SAMPLES

35DNA IN SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil samples for 35DNA.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard soil is 0.525 to 10.5 ug/g.

B. SENSITIVITY

The normalized response (integrator counts) at the natural soil detection limit designated in Section 1(C) is listed below:

Analyte	Integrator Counts	Nanograms	
35DNA	167,078	550	

The normalized response (integrator counts) at the standard soil detection limit designated in Section 1(C) is listed below:

Analyte	Integrator Counts		Nanograms	
35DNA	79.742	•	263	

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is 1.1 ug/g. The detection limit in standard soil is 0.53 ug/g.

D. INTERFERENCES

This method may be subject to interferences from nonvolatile, water soluble organic compounds which absorb light at 395 nm and are extractable from water with methylene chloride. Interferences are minimized by Florisil® Sep-Pak® cleanup.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 20 extracts in an 8-hour day. One analyst can perform approximately six extractions in an 8-hour day.

2. CHEMISTRY

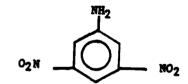
A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS)
REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
35DNA	3,5-dimitroaniline,	618-87-1
	1-amino-3,5-dinitrobensene	
	3,5-dinitrobenzenamine	

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

			Acid	
Analyte	Formula	Melting Point (°C)	Dissociation Constant	
35DNA	C6 H504N3	160-162	$pk_a = 0.23$	

Chemical Structure



C. CHEMICAL REACTIONS

35DMA reacts as a weak base.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

- 1. Detector: Perkin-Elmer LC-75 variable-wavelength detector ($\lambda = 395 \text{ nm}$)
- 2. Column: Ultrasphere ODS (4.6-mm ID x 25 cm)

Particle size: 5 um

3. Flow Rate/Mobile Phase: 1 ml/min

30% water/70% methanol

- 4. Temperature: 25°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Time: 5.6 minutes

C. HARDWARE/GLASSWARE

- 1. 250-ml glass separatory funnels with stoppers (6);
- 2. Short-stemmed glass funnels (6);
- 3. 500-ml K-D evaporative flasks (6);
- 4. 10-ml graduated K-D receivers (6);
- 5. 3-ball Snyder columns (6);
- 6. 2-ball micro-Snyder columns (6);
- 7. 10-ml graduated centrifuge tubes (6);
- 8. 5-ml glass syringes with Luer-lock attachments (6);
- 9. 50-ml centrifuge tubes with Teflon -lined screw caps; and
- 10. 100-ml graduated cylinder (1).

D. CHEMICALS

- 1. Nanograde methylene chloride;
- 2. HPLC-grade methyl alcohol--J.T. Baker Company;
- 3. HPLC-grade water--J.T. Baker Company;
- 4. Anhydrous sodium sulfate, reagent grade;
- 5. Glass wool;
- 6. Teflon boiling chips;
- 7. Florisil® Sep-Paks®--Waters Associates;

4. STANDARDS

A: CALIBRATION STANDARDS

- The 35DNA stock calibration standard (1.05 mg/ml) is prepared by weighing 10.5 mg of 35DNA into a 10-ml volumetric flask, dissolving the 35DNA in a few ml of methanol, and diluting with methanol to the mark.
- 2. Prepare a series of working calibration standards by making dilutions of the stock calibration standard. Dilute with 50% methanol/50% water as follows:

Working Calibration Standard	Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)	
A	Stock Calibration Standard	5	25	
В	Standard A	1	10	
С	Standard A	0.5	10	
D	Standard A	0.2	10	
E	Standard A	1.0	10	
F	Standard A	0.05	10	
G	Standard D	1	10	

Working Calibration Standard	Concentration (ug/ml)
A	210
В	21.0
С	10.5
D	4.2
E	2.1
F	1.05
G	0.42

B. CONTROL SPIKES

- 1. Use Working Calibration Standard A as the control spike solution.
- 2. Weigh out 20.0 g of soil into a 50-ml centrifuge tube.
- 3. Pipet a known amount of the control spike solution (dissolved in a sufficient volume of HPLC-grade water to just wet the soil) into the centrifuge tube. The quantity spiked should be selected to provide a concentration of 0.5 to 10.0 times the detection limit.

Volume of Control Spike Solution (ml)	Concentration of Spiked Water (ug/L)
0.05	0.525
0.10	1.05
0.20	2.10
0.50	5.25
1.00	10.5

4. Shake the sample to mix and let air dry for 1 hour.

5. PROCEDURE

A. SAMPLE PREPARATION

- 1. Let soil sample air dry on dull side of aluminum foil until it can be sieved through a 30-mesh sieve.
- 2. Sample the soil by quartering and place 20.0 g into a 50-ml centrifuge tube.

B. EXTRACTION

- Add 35 ml of HPLC-grade water to the centrifuge tube and shake for 3 minutes. Centrifuge the sample at approximately 3,000 rpm for 15 minutes and decant the water layer into a 250-ml separatory funnel.
- 2. Repeat Step 1 twice more and combine the extracts.
- 3. Extract the water extracts sequentially with three 80-ml portions of methylene chloride using 2-minute shake times

- and 10-minute separation times. Centrifuge to separate any resulting emulsions.
- 4. Pass the methylene chloride extracts through a glass funnel containing approximately 20 g of sodium sulfate and a glass wool plug. Collect the extracts in a K-D apparatus equipped with a 10-ml receiver. Rinse the sodium sulfate with approximately 20 ml of methylene chloride. Add a Teflon boiling chip to the extract, and attach a three-ball Snyder column to the apparatus.
- 5. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath, immersing the receiver nearly up to the joint. The balls should chatter actively at the proper evaporation rate.
- 6. Let the apparent volume of extract decrease to approximately 2 ml, then remove from the bath, and let cool. Remove the receiver from the K-D apparatus and add methylene chloride to raise the volume to approximately 5 ml.
- 7. Attach a Florisil® Sep-Pak® to a 5-ml syringe and transfer the extract into the syringe. Pass the entire extract through the Sep-Pak® at a rate of approximately 5 ml/min and discard the eluate. Rinse the receiver with an additional 1 ml of methylene chloride, pass the rinses through the Sep-Pak®, and discard the eluate.
- 8. Elute the analyte from the Sep-Pak® with 5 ml of 5% methanol in methylene chloride and collect in the original receiver.
- 9. Add approximately 2 ml of methanol and a fresh Teflon® boiling chip to the receiver, and attach the two-ball micro-Snyder column. Concentrate to approximately 2 ml on a 90°C water bath. Add an additional 10 ml of methanol and reconcentrate to approximately 2 ml. Repeat this process once more by adding 10 ml of methanol and concentrating to a volume of approximately 2.0 ml.
- 10. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube, rinsing

the receiver quantitatively with 2.0 ml of HPLC-grade water. Raise the extract volume to exactly 10.0 ml in the centrifuge tube with HPLC-grade water.

- 11. Transfer to a 2-ml septum-sealed vial for storage at 4°C.
- 12. The extract is now ready for HPLC analysis.

B. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample using the conditions given in Section 3(B).
- 3. Measure the response of the 35DNA peak.

6. CALCULATIONS

- A. Prepare a standard curve of concentration (ug/ml) of standard versus peak area counts.
- B. Determine the concentration of 35DNA according to the following formula:

Concentration (ug/g) =
$$\frac{(A)(V_t)}{W_8}$$

where: A = Concentration (ug/ml) of analyte found in the sample extract by comparison with the appropriate standard curve,

 V_t = Volume of total extract (ml), and

Wg = Weight of initial sample extracted (g).

7. REFERENCES

None found.

8. DATA

See attached data sheets.

Chromatogram of the Extract of a Natural Soil Sample Spiked with 35DNA at 2.1 ug/g

3.5 DINITROANILINE.STD.SOIL(UG/G)

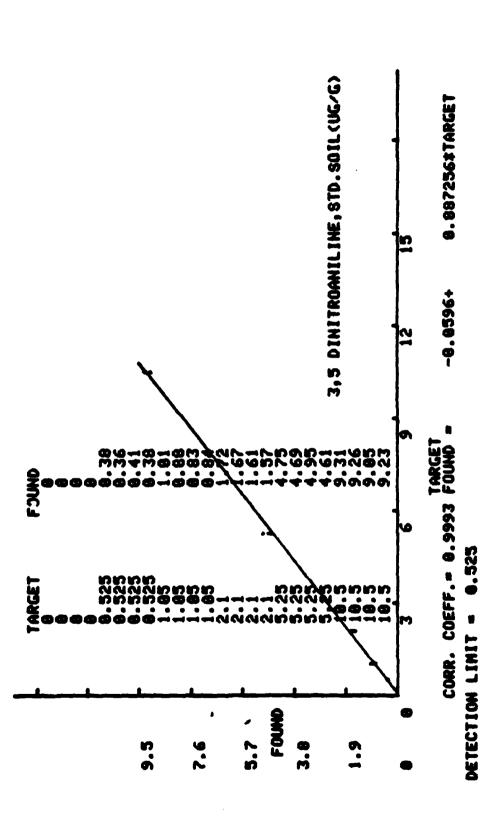
TARGET CONCENTRATION	1	DAY 2	3	4	
9.0000	0.0000	G.0000	0.0000	0.0000	
0.525	0.380	0.360	0.410	0.380	
1.05	1.01	0.880	0.830	0.840	
2.10	1.72	1.67	1.61	1.57	
5 • 25	4•75	4 • 6 9	4 • 95	4.61	
10.5	9.31	9.26	9.05	9.23	

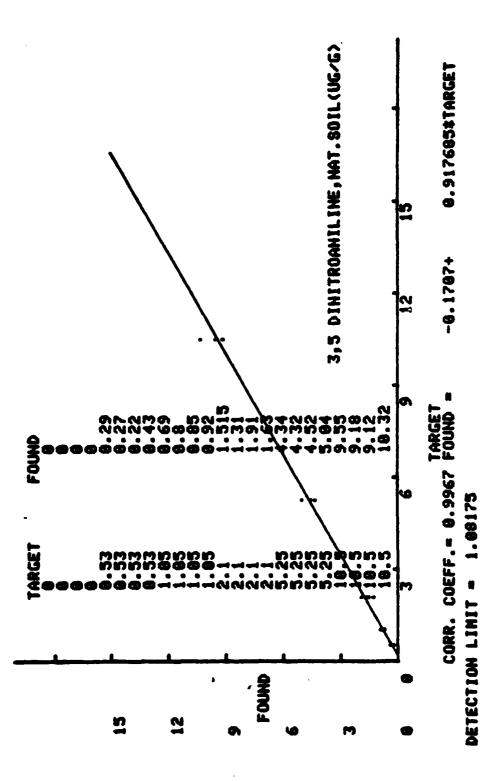
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0 • 0 0 0 0	0.000	0.0000	0.000
0.525	0.382	0.0206	5.39	-27.1429
1.05	ŭ•8 9 0	0.0829	9.31	-15.2381
2.10	1.64	0.0660	4.02	-21.7857
5.25	4.75	0.145	3.06	-9.5238
10.5	9•21	0.113	1.23	-12.2619

3.5 DINITROANILINE.NAT.SOIL(UG/G)

TARGET . CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	0.0000	
0.530	0.290	0.270	0.220	0.430	
1.05	0.690	0.800	0.850	0.920	
2.19	1.51	1.31	1.91	1.63	
5.25	4.34	4.32	4.52	5.04	
10.5	9.55	9.18	9.12	10.3	

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.000
0.530	0.302	0.0900	29.7	-42.9245
1.05	0.815	0.0968	11.9	-22.3810
2.10	1.59	0.250	15.7	-24.2262
5.25	4.55	0.336	7.37	-13.2381
10.5	9.54	0.552	5.79	-9.1191





TDGCL IN WATER SAMPLES

TDGCL IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for TDGCL.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural water is 40 to 800 ug/L.

B. SENSITIVITY

The normalized response (peak height in mm times attenuation) at the detection limit designated in Section 1(C) is 4,736 mm for 2,975 ng of TDGCL.

C. DETECTION LIMIT

The detection limit in natural water, calculated according to Hubaux and Vos (1970), is 119 ug/L.

D. INTERFERENCES

This method may be subject to interferences from highly watersoluble compounds which absorb light at 215 nm. Because of the
polarity of glycols, it is not possible to concentrate them
quantitatively by solvent extraction; therefore, their
determination must be carried out in the squeous phase. TDGCL
is concentrated by boiling off the water taking advantage of
its high boiling point (165°C). A cleanup of the sample is
acheived by column chromatography on Amberlite® XAD-7 resin
which removes some of the UV-absorbing interferents occuring in
natural surface waters. These interferents have not been
observed in ground waters examined and seem to be generated by
hydrolysis of high molecular weight substances (possibly humic
and fulvic acids) occurring in surface waters.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 6 extracts in an 8-hour day. One analyst can perform 6 extractions and boildowns in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCIATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
TDGCL	2,2'-Thiodiethanol	111-48-8
	Bis (b-hydroxyethyl)	
	Sulfide	

B. PHYSICAL AND CHEMICAL PROPERTIES

		Melting	Boiling	Density	
Analyte	Formula	Point (°C)	Point (°C)	(g/m1)	
TDGCL	C4H10O2S	-10	165	1.1819	

C. CHEMICAL REACTIONS

None found.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 gradient liquid chromatograph (HPLC) equipped with a Perkin-Elmer LC-75 variable-wavelength, UV-visible detector and interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC PARAMETERS

- 1. Detector: Perkin-Elmer LC-75 variable-wavelength detector (λ = 215 nm)
- 2. Column: Ultrasphere ODS (4.6-mm ID x 25 cm)
 Particle Size: 5 um

- 3. Flow Rate and Mobile Phase: 1 ml/min of phosphate buffer solution, pH = 3.0
- 4. Temperature: 22°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Time: 11.8 minutes

C. HARDWARE/GLASSWARE

- 1,000-ml separatory funnel (Teflon[®] or glass) (6);
- 2. 600-ml beaker (6);
- 3. 10-cm diameter watch glass (6);
- 4. 50-ml beaker (6); and
- 5. 10-ml graduated centrifuge tubes (6).

D. CHEMICALS AND REAGENTS

- 1. Nanograde methylene chloride--J.T. Baker Company;
- 2. HPLC-grade acetonitrile--J.T. Baker Company;
- 3. HPLC-grade water--J.T. Baker Company;
- 4. 6N sodium hydroxide;
- 5. 6N sulfuric acid; and
- 6. Phosphate buffer--5 g of NH₄H₂PO₄ and 1 ml of 85% H₃PO₄ in HPLC-grade water (pH = 3.0).

4. STANDARDS

A. CALIBRATION STANDARDS

- 1. Prepare a stock calibration standard (10 mg/ml) by weighing out 100 mg of TDGCL into a single 10-ml volumetric flask and diluting to volume with HPLC-grade water. Wrap the flask in foil and store at 4°C.
- Prepare a dilute stock calibration standard by pipetting 1 ml of the stock calibration standard into a 50-ml volumetric flask and diluting to volume with HPLC-grade water.

3. Prepare the working calibration standards by making dilutions of the dilute stock calibration standard using the appropriate volumetric flasks and HPLC-grade water as follows:

Working Calibration Standard	Concentration (ug/ml)	Volume of Dilute Stock Taken (m1)	Final Volume (ml)
В	4.0	1	50
C	8.0	2	50
D	16	4	50
E	40	10	50
F	80	20	50

B. CONTROL SPIKES

- 1. Prepare the stock control spike standard by weighing 100 mg of TDGCL, transferring to a 10-ml volumetric flask, and diluting to volume with HPLC-grade water.
- 2. Prepare the working control spike standard by pipetting 2 ml of the stock control spike standard into a 100-ml volumetric flask and diluting to volume with HPLC-grade water.
- 3. Pipet known amounts of the working control spike standard into standard water. The quantity spiked should be selected to provide a concentration range of 0.5 to 10 times the detection limit.
- 4. Determine the accuracy and detection limit by pipetting the following amounts of the working control spike standard into 500 ml of standard water and analyzing according to the procedure outlined in Section 5:

Volume of Working Control Spike Standard Spiked (ml)	Concentration of TDGCL (ug/L)
0	0
0.100	40
0.200	80
0.400	160
1.00	400
2.00	800

5. PROCEDURE

A. BOILDOWN AND COLUMN CLEANUP

- 1. It is important that the following procedures be performed in one 8-hour day.
- 2. Measure 500 ml of the water sample into a 1-L beaker.
- 3. Add a boiling chip (Teflon®) and concentrate the sample by boiling the water on a hot plate to a volume of 50 ml. The boildown time should be as rapid as possible and should not exceed 2.5 hours.
- 4. Adjust the pH of the sample to 3 and transfer the sample to a column (20 cm x 1 cm) packed with Amberlite® XAD-7 resin. The resin is prepared by shaking 50 g of resin with 100 ml of methanol for 15 minutes on a wrist-action shaker. The methanol is decanted, and the operation is repeated sequentially with three 100-ml portions of methanol followed by four 100-ml portions of HPLC-grade water. The column is slurry packed in water. The column flow rate is controlled at a rate of 1 ml/min, and the eluate is collected in a 250-ml beaker. The column is rinsed with 50 ml of HPLC-grade water into the same beaker to give a total volume of approximately 100 ml.
- 5. The volume of the solution is further reduced by boiling to less than 25 ml, and then quantitatively transferred with rinsing into a 50-ml beaker.
- 6. The volume of the solution is then reduced to less than 5 ml by boiling on a hot plate.
- 7. Transfer the solution into a 10-ml graduated centrifuge tube, rinsing quantitatively with HPLC-grade water. Dilute to the 5 ml mark with HPLC-grade water.
- 8. Filter the sample through a 0.45-um filter and transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
- 9. The solution is now ready for chromatography by HPLC.

B. CALIBRATION

- Inject the Working Calibration Standards B, C, D, E, and F and a blank singly at the beginning of the analytical run. Inject the Working Calibration Standard D at the conclusion of the analytical run to verify constant instrument response.
- 2. Plot the normalized peak heights versus nanograms injected of each standard to obtain a working curve.

C. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample according to the conditions given in Section 3(B).
- 3. Measure the response of the sample for the component of interest.

6. CALCULATIONS

Determine the concentration of TDGCL according to the following formula:

Concentration
$$(ug/g) = \frac{(A)(V_t)}{(V_i)(V_g)}$$

where: A = Nanograms of TDGCL found in the sample by comparison with the appropriate standard curve,

 V_t = Final volume of solution (ml),

Vg = Volume of initial sample extracted (ml), and

V_i = Volume injected (ml).

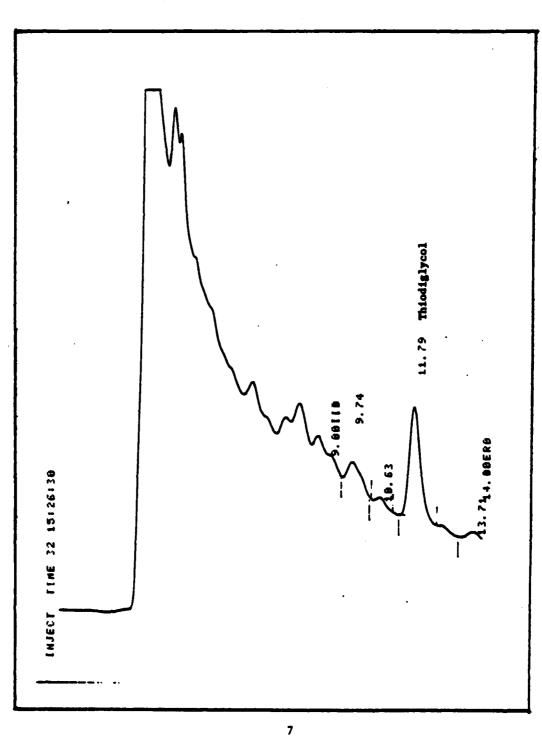
7. REFERENCES

None found.

8. DATA

See attached data sheets.

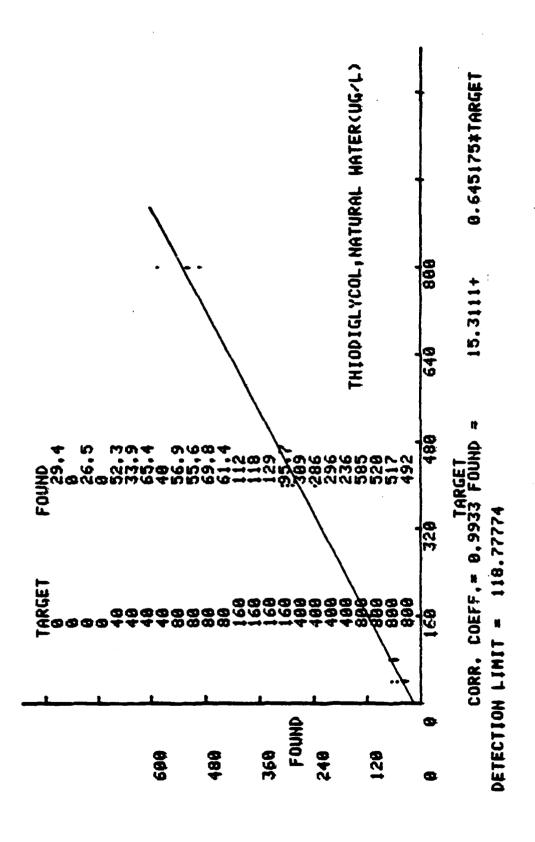
Figure 1 Chromatogram from the analysis of 500 ml of a surface water sample spiked at 400 ppb.



THIODIGLYCOL NATURAL WATER (UG/L)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0500	29•4	0.0000	26•5	0.0000	
40.0	52.3	33.9	65.4	40.0	
80.0	56.9	55.6	69.8	61.4	
160	112	118	129	95.7	
400	309	286	296	236	
800	585	520	517	492	

TARGET CONCENTRATION	AVERAGE Found value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	14.0	16.2	116	0.0000
40.0	47.9	14.0	29.1	19.7
80.0	60.5	6.42	10.5	-23.8438
160	114	13.9	12.2	-28.9531
4 C E	282	31.9	11.3	-29.5625
ecc	529	39.7	7.51	-33.9375



TDGCL IN SOIL SAMPLES

TDGCL IN NATURAL SOIL SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental soil and sediment samples for TDGCL.

A. TESTED CONCENTRATION RANGE The tested concentration range in natural soil is 1 to 20 ug/g.

B. SENSITIVITY

The normalized response (peak height in mm x attenuation) at the natural soil detection limit is 1,056 mm for 1.02 ug of TDGCL.

C. DETECTION LIMIT

The detection limit in natural soil, calculated according to Hubaux and Vos (1970), is 2.2 ug/g.

D. INTERFERENCES

This method may be subject to interferences from highly water-soluble compounds which absorb light at 215 nm. Because of the polarity of the glycols, it is not possible to extract them from soil quantitatively by organic solvent extraction; therefore, their determination is performed by extraction of the soil with water. TDGCL is concentrated by evaporation of the water by boiling, taking advantage of its high boiling point of 165°C. A cleanup of the sample is achieved by column chromatography of the acidified aqueous extract on Amberlite® XAD-7 resin. Partial sample cleanup is also achieved by using an acidic aqueous extraction; only highly water-soluble substances will be extracted. After column chromatography, the water is neutralized and boildown proceeds.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 8 extracts in an 8-hour day. One analyst can perform 8 extractions and boildowns in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS)
REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
TDGCL	2,2'-Thiodiethanol	111-48-8
	Bis (B-hydroxyethyl) Sulfide	

B. PHYSICAL AND CHEMICAL PROPERTIES

Analyte	Formula	Melting Point (°C)	Boiling Point (°C)	Density (g/ml)
TDGCL	C4H10O2S	-10	165	1.1819

C. CHEMICAL REACTIONS
None.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 gradient liquid chromatograph (HPLC) equipped with a Perkin-Elmer LC-75 variable-wavelength UV-visible detector and interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector (λ = 215 nm)

- 2. Column: Ultrasphere ODS (4.6-um ID x 25 cm)
 Particle Size: 5 um
- 3. Flow Rate and Mobile Phase: 1 ml/min phosphate buffer solution
- 4. Temperature: 22°C
- 5. Injection Volume: 250 ul, fixed loop
- 6. Retention Time: 12.5 minutes

C. HARDWARE/GLASSWARE

- 1. 250-ml Erlenmeyer flasks with screw caps (8);
- 2. 600-ml beakers (8);
- 3. 10-cm-diameter watch glasses (8);
- 4. 50-ml beakers (8);
- 5. 10-ml graduated centrifuge tubes (8);
- 6. Burrell Model 75 wrist-action shaker (1); and
- 7. Glass chromatography columns (20 cm x 1-cm ID) (8).

D. CHEMICALS AND REAGENTS

- 1. HPLC-grade water--J.T. Baker Company;
- 2. 6N sodium hydroxide;
- 3. 6N sulfuric acid; and
- 4. Phosphate buffer--5.75 g of NH₄H₂PO₄ and 1 ml of 85% H₃PO₄ in 1 L of HPLC-grade water (pH = 3.0).

4. STANDARDS

A. CALIBRATION STANDARDS

- Prepare a stock calibration standard (10.13 mg/ml) by weighing out 101.3 mg of TDGCL into a single 10-ml volumetric flask and diluting to volume with HPLC-grade water. Wrap the flask in foil and store at 4°C.
- Prepare a dilute stock calibration standard by pipetting l ml of the stock calibration standard into a 50-ml volumetric flask and diluting to volume with HPLC-grade water.

3. Prepare the working calibration standards by making dilutions of the dilute stock calibration standard using the appropriate volumetric flasks and HPLC-grade water as follows:

Working Calibration Standard	Volume of Dilute Stock Used (ml)	Final Volume (ml)	Concentration (ug/mL)
В	0.5	50	2.03
C	1	50	4.05
D	2	50	8.10
E	5	50	20.3
F	10	50	40.5

B. CONTROL SPIKES

- Prepare the stock control spike standard (10.13 mg/ml) by weighing 101.3 mg of TDGCL, transferring to a 10-ml volumetric flask, and diluting to volume with HPLC-grade water.
- 2. Prepare the working control spike standard by pipetting l ml of the stock control spike standard into a 50-ml volumetric flask and diluting to volume with HPLC-grade water.
- 3. Pipet known amounts of the working control spike standard into standard soil. The quantity spiked should be selected to provide a concentration range of 0.5 to 10 times the detection limit.
- 4. Determine the accuracy and detection limit by pipetting the following amounts of the working control spike standard into 10 g of soil and analyzing according to the procedure outlined in Section 5:

Volume of Working Control Spike Standard Spiked (ml)	Concentration of TDGCL (ug/g)
0	0
0.05	1.0
0.1	2.0
0.2	4.0
0.5	10
1.0	20

5. PROCEDURE

A. SAMPLE PREPARATION

1. The soil sample should be air dried on the dull side of aluminum foil and then sieved through a 30-mesh sieve.

B. EXTRACTION

- 1. It is important that the following procedures be performed in one 8-hour day.
- 2. Measure 10 g of sieved, dried soil or wet sediment into a tared 250-ml centrifuge tube with screw cap.
- 3. Add 50 ml of HPLC-grade water and adjust the pH to 3 using $6N\ H_2SO_4$.
- 4. Cap the tube, shake vigorously by hand for 5 minutes, and centrifuge at 2,250 rpm for 15 minutes.
- 5. The supernatant liquor is decanted and collected in a 250-ml beaker.
- 6. Step 5 is repeated twice. After collecting all supernatant water, the pH is adjusted to 3 using 6N H_2SO_4 .
- 7. Quantitatively transfer contents of the 250-ml beaker to a glass column (20 cm x 1-cm ID) packed with Amberlite® XAD-7 resin. (The resin is previously prepared by shaking 50 g of the resin with 100 ml of methanol for 15 minutes on a wrist-action shaker. The methanol is decanted, and the operation is repeated sequentially with three 100-ml portions of methanol followed by four 100-ml portions of

HPLC-grade water. The column is slurry-packed in water. The resin must be cleaned after each sample and may be reused a total of three times.) Pass the sample through the column at maximum flow (approximately 4 ml/min), and collect in a 300-ml beaker. The column is rinsed and eluted to dryness with 50 ml of HPLC-grade water into the same beaker to give a total volume of approximately 250 ml.

- 8. Add a Teflon[®] boiling chip, reduce the volume of the water to less than 25 ml by boiling on a hot plate, and quantitatively transfer the solution into a 50-ml beaker.
- 9. Reduce the volume of the solution to less than 5 ml by boiling on a hot plate.
- 10. Transfer the solution into a 10-ml graduated centrifuge tube, rinsing quantitatively with HPLC-grade water, and dilute to the 5-ml mark with the same water.
- 11. Filter the sample through a 0.45-um filter, and transfer to a 5-ml, amber, septum-sealed vial for storage at 4°C.
- 12. The solution is now ready for HPLC analysis.

C. CALIBRATION

- Inject Working Calibration Standards B, C, D, E, and F and a blank singly at the beginning of the analytical run. Inject Working Calibration Standard D at the conclusion of the analytical run to verify constant instrument response.
- 2. Plot the normalized integrator response versus ng/ul of each standard to obtain a working curve.

D. ANALYSIS

- 1. Inject 250 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample according to the conditions given in Section 3(B).
- 3. Measure the response of the sample for the TDGCL peak.

6. CALCULATIONS

Determine the concentration of TDGCL according to the following formula:

Concentration (ug/g) = $\frac{(A)(V_t)}{(V_i)(V_g)}$

where: A = Weight of TDGCL found in the sample by comparison with the appropriate standard curve (ng),

Vt = Final volume of solution (ml),

 V_{g} = Weight of initial sample extracted (g), and

V_i = Volume injected (ul).

7. REFERENCES

None found.

8. DATA

See attached data sheets.

Chromatogram of Natural Soil Extract Spiked with Thiodiglycol

INJECT FIME 11 11:31:37
PT= 386.

- 00118

12. 21

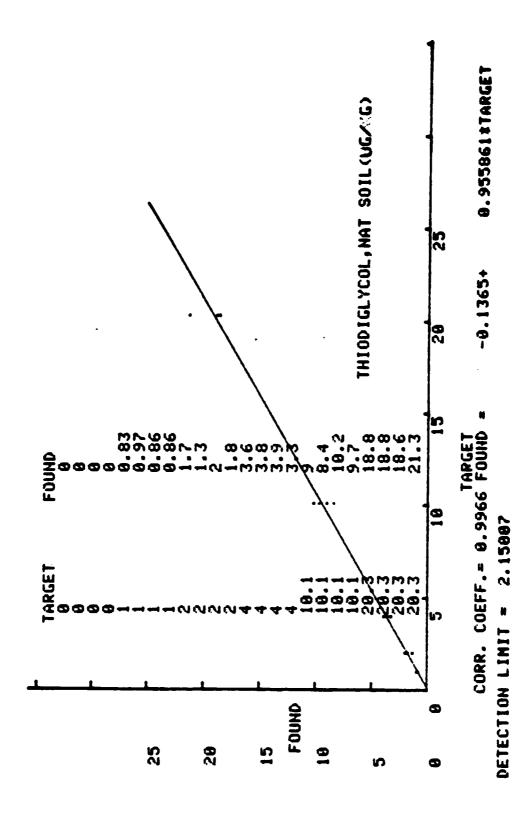
13. 89 THIODICLYCOL

SPIRE PRODUCTOR

THIODIGLYCOL.MAT SOIL (UG/ G)

TARGET CONCENTRATION	1	DAY 2	3	4	
0.000	0.0000	0.0000	0.0000	0.0000	
1 • 9 3 8	0.830	0.970	0.860	0.860	
2.00	1.70	1.30	2.60	1.60	•
4.00	3.60	3.80	3.90	3.30	
16.1	9.06	8 - 40	10.2	9.70	
20.3	18.8	18+8	18.6	21.3	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT I NACCURACY
0.0031	0.0000	9.0C00	0.0000	0.0000
1.00	0.880	0.0616	7.01	-12.0000
2 • 3 €	1.70	3.294	17.3	-15.0000
4.75	3.65	3.265	7.25	-8.7500
10.1	9.32	0.789	8 • 46	-7.6733
20.3	19.4	1.29	6.54	-4.5567



ì.

HPLC SCREEN OF WATER SAMPLES FOR
NITROSUBSTITUTED MUNITION COMPOUNDS AND PAHS

HPLC SCREEN OF WATER SAMPLES FOR NITROSUBSTITUTED MUNITION COMPOUNDS AND PAHS

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for nitrosubstituted munition organics and PAHs by HPLC. Detector ratios are employed to provide data for the qualitative identification of the analytes. Quantitative documentation was performed on the analytes listed in Table I using the detectors and wavelengths indicated. Documentation was performed for samples with and without silica-gel chromatography cleanup.

A. TESTED CONCENTRATION RANGE

The tested concentration range in natural and standard water for each analyte is listed in Table 2.

B. SENSITIVITY

The normalized responses (integrator peak height counts) at the standard water detection limits (without silica-gel cleanup) designated in Section 1(c) are listed in Table 3.

The normalized responses (integrator peak height counts) at the natural water detection limits (without silica-gel cleanup) designated in Section 1(c) are listed in Table 4.

C. DETECTION LIMIT

The detection limits (DL) in standard and natural water, calculated according to Hubaux and Vos (1970), are listed in Table 5.

The detection limits for standard water samples cleaned by silica-gel chromatography are listed to indicate the quality

Table 1. Analytes and Quantitative Detection Methods

Analyte	Quantitative Detection Method
нмх	UV (230 nm)
RDX	UV (230 nm)
135TNB	UV (230 nm)
1 3 DNB	UV (254 nm)
35DNP	UV (254 nm.)
246TNT	UV (254 nm)
26DNT	UV (230 nm)
24DNT	UV (254 nm.)
Naphthalene	UV (280 nm)
Acenaphthylene	UV (280 nm)
Acenaphthene	UV (280 nm)
Phenanthrene	UV (280 nm)
Anthracene	UV (254 nm)
Fluoranthene	UV (280 nm)
Pyrene	UV (280 nm)
Chrysene	UV (280 nm)
Benzo(b)fluoranthene	Fluorescence (λ ex 290 nm, λ em > 350 nm)
Benzo(k)fluoranthene	Fluorescence (λ ex 290 nm, λ em > 350 nm)
Benzo(a)pyrene	UV (254 nm)
Indeno(1,2,3-cd)pyrene	UV (280 nm)

Table 2. Tested Concentration Ranges in Natural and Standard Water

Analyte	Concentration Range (ug/L)
нмх	2.0 to 40
RDX	2.0 to 48
135TNB	1.9 to 38
1 3 DNB	2.2 to 44
35DNP	3.6 to 73
246TNT	2.0 to 40
26DNT	2.3 to 46
24DNT	2.0 to 40
Naphthalene	4.1 to 82
Acenaphthylene	6.6 to 131
Acenaphthene	1.5 to 48
Phenanthrene	1.5 to 30
Anthracene	1.2 to 24
Fluoranthene	0.4 to 8.0
Pyrene	1.9 to 38
Chrysene	0.10 to 2.0
Benzo(b)fluoranthene	0.10 to 2.1
Benzo(k)fluoranthene	0.21 to 4.1
Benzo(a)pyrene	0.22 to 4.5
Indeno(1, 2, 3-cd)pyrene	0.28 to 5.8

Table 3. Sensitivity at Standard Water Detection Limits Without Cleanup

Analyte	Integrator Peak Height Counts	Nanograms
HMX	2,354	74
RDX	466	30
135TNB	985	30
13DNB	507	31
35DNP	605	56
246TNT	226	29
26DNT	392	46
24DNT	299	29
Naphthalene	1,406	137
Acenaphthylene	514	109
Acenaphthene	272	49
Phenanthrene	593	26
Anthracene	1,786	11
Fluoranthene	185	4.3
Pyrene	241	38
Chrysene	78	2.0
Benzo(b)fluoranthene	633	8.3
Benzo(k)fluoranthene	520	5.8
Benzo(a)pyrene	323	4.2
<pre>Indeno(1,2,3-cd)pyrene</pre>	164	9.1

Table 4. Sensitivity at Natural Water Detection Limits Without Cleanup

Analyte .	Integrator Peak Height Counts	Nanograms
HMX	1,550	48
RDX	464	30
135TNB	357	32
13DNB	871	53
35DNP	1,869	173
246TNT	265	34
26DNT	307	36
24DNT	419	40
Naphthalene	1,047	103
Acenaphthylene	661	140
Acenaphthene	279	51
Phenanthrene	526	24
Anthracene	3,959	23
Fluoranthene	305	8
Pyrene	218	33
Chrysene	60.2	1.6
Benzo(b)fluoranthene	200	2.8
Benzo(k)fluoranthene	619	7.0
Benzo(a)pyrene	722	9.8
Indeno(1,2,3-cd)pyrene	222	12

Table 5. Detection Limits in Standard and Natural Water*

Analyte	Standard Water (ug/L)	Standard Water (After Silica-Gel Cleanup) (ug/L)	Natural Water (ug/L)
нмх	15	11	10
RDX	6	8	6
135TNB	6	6	6
13DNB	6	8	11
35DNP	11	28	34
246TNT	6	11	7
26DNT	9	12	7
24DNT	6	7	7
Naphthalene	27	33	21
Acenaphthylene	22	35	28
Acenaphthene	10	12	10
Phenanthrene	5	5	5
Anthracene	2	5	5
Fluoranthene	0.9	1	2
Pyrene	8	6	7
Chrysene	0.4	0.5	0.3
Benzo(b)fluoranthene	2	0.3	0.6
Benzo(k)fluoranthene	1	0.8	1
Benzo(a)pyrene	0.8	0.9	2
Indeno(1,2,3-cd)pyrene	2	2	2

^{*} Calculated according to Rubaux and Vos, 1970.

of the cleanup technique for each analyte. The detection limits listed for standard water without silica-gel cleanup are the actual detectability criteria to be used for the analytes. The silica-gel cleaned extracts are analyzed only when a suspected target analyte is detected in the noncleaned extracts and when large unresolved background interferences prevent adequate quantitation.

D. INTERFERENCES

This method may be subject to interferences from organic compounds which are extractable from acidic water with methylene chloride. The qualitative analysis via detector ratios should be sufficient to prevent misidentification of unknown peaks. The silica-gel cleanup step provides a mechanism for eliminating possible interferences from highly polar compounds such as fatty acids and carboxylic acids which may be present in the extract. An unidentified fluorescence peak occurred at 25.7 minutes during the standard water documentation. This peak did not appear in either the standard or natural water documentation and did not display a measurable absorbance at 254 or 230 nm. Therefore, the compound should not interfere in the method.

Several compounds were not fully documented because of co-elution with another analyte under the screen conditions, chemical instability, or experimental interferences. However, the retention times and detector ratios for most of these compounds were determined and are listed in Table 6.

E. ANALYSIS RATE

After instrument calibration, which requires approximately 6 hours, one analyst can analyze four extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

Table 6. Retention Times and Detector Ratios for Compounds which were not Adequately Resolved and/or Documented

Analyte	Problem	Retention Time (minutes)	Detector Ratio (Wavelength/ Wavelength) (nm)
Tetryl	Co-elutes with nitrobenzene under certain column conditi	(24.4)* 29.5	3.14 (230/280)
NB	Co-elutes with tetryl under certain column conditions	24.8	0.701 (230/280)
ATNBA	Unstable	14.3	2.8 (230/280)
12DNT	Internal standar not documented		0.8518 (230/280)

^{*} The peak for tetryl undergoes significant shifts in retention time depending on the condition of the ODS column used in the system.

Under the preliminary column conditions for documentation, tetryl interfered with nitrobenzene (RT = 24.4 min). After a new ODS column was installed, the RT for tetryl had shifted (RT = 29.5 min).

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS)
REGISTRY NUMBERS
The alternate nomenclature and CAS registry numbers for the

B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTES

The physical and chemical properties for the analytes of interest are listed in Table 8.

analytes of interest are listed in Table 7.

C. CHEMICAL REACTIONS

Caution should be used in handling all of these compounds, especially the standard materials. All of the nitrosubstituted compounds are either explosives or breakdown products of explosives. The PAHs are known carcinogens, teratogens, and mutagens.

3. APPARATUS

A. INSTRUMENTATION

The HPLC instrumentation (see Figure 1) is a gradient elution system with two columns connected in series with a variable-wavelength, UV-visible absorbance detector; a 254-nm absorbance detector; and a fluorescence detector.

An Altex Model 322 dual-pump liquid chromatograph was used as the gradient pumping system. After mixing, the elution solvent was passed through a guard column (5 cm by 4.6 mm) packed with silica gel (Fisher, 60 to 200 mesh) to presaturate the mobile phase with silica and therefore extend column life. The mobile phase was then passed through a 0.25-um filter to remove entrained particulates.

An Altex Model 500 autosampler was used as the injection system, and a guard column (5 cm by 4.6 mm) packed with Pelliguard LC-CN pellicular packing (40 um) was present in the system. Both the

Table 7. Alternate Nomenclature and CAS Registry Numbers

Analyte	Alternate Nomenclature	CAS Registry Number
нмх	Cyclotetramethylenetetranitramine Octahydro-1,3,5,7-tetrazocine 1,3,5,7-Tetranitro-1,3,5,7- tetrazacyclooctane Octogen	2691-41-0
RDX	Cyclotrimethylenetrinitramine Hexogen, T-4, Cyclonite, Hexahydro- 1,3,4-trinitro-s-triazine	121 -84 -4 -
135TNB	sym-Trinitrobenzene benzite	25377 -32- 6
13DNB	m-Dinitrobenzene	99-65-01
35DNP		586-11-8
246TNT	<pre>sym-Trinitrotoluene 1-methy1-2,4,6-trinitrobenzene trotyl; Tolit, Trilit</pre>	118-96-7
26DNT		606-20-2
24DNT		121-14-2
Naphthalene		91-20-3
Acenaphthylene		208-96-8
Acenaphthene	Naphthyleneethylene	82-32-9
Phenanthrene		85-01-8
Anthracene	p-Naphthalene	120-12-7
Fluoranthene	1,2-Benzacenaphthene Idryl	86-73-7
Pyrene	Benzo(d,e,f)phenanthrene	129-00-0
Chrysene	1,2-Benzophenanthrene Benzo(a)phenanthrene	218-01 <i>-</i> 9

Table 7. Alternate Nomenclature and CAS Registry Numbers (Continued, Page 2 of 2)

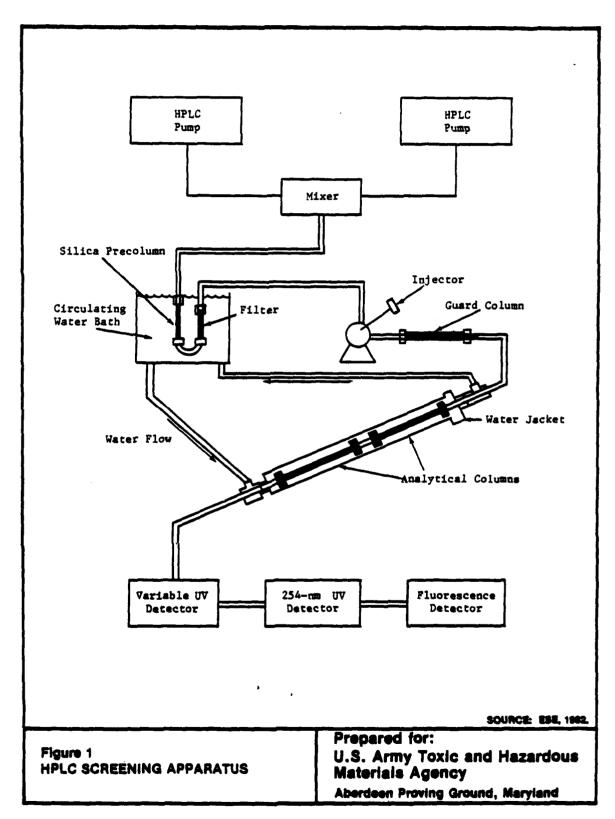
Analyte	Alternate Nomenclature	CAS Registry Number
Benzo(b)fluoranthene	3,4-Benzofluoranthene Benz(e)acephenanthrylene	205-99-2
Benzo(k)fluoranthene	11,12-Benzofluoranthene	207-08-9
Benzo(a)pyrene	3,4-Benzopyrene	50-32-8
Dibenzo(a,h)anthracene	1,2:5,6-Dibenzanthracene	53-70-3
Indeno(1,2,3-cd)pyrene	<pre>Indeno(1,2,3)pyrene 2,3-o-Phenylenepyrene</pre>	193-39-5

Table 8. Physical and Chemical Properties

Analyte	Formula	Melting Point (°C)	Boiling Point (°C)	Density (g/ml @ 20°C)
HMX	C4H8N8O8	276		1.77-1.96*
RDX	C3H6N6O6	204.1		1.816
135TNB	C6H3N2O4	122	t	1.69
13DNB	C6H4N2O4	90	302	1.57
35DNP	C6H4N2O2	126		1.702
246TNT	C7H5N3O6	80	280†	1.65
26DNT	C7H5N2O4	66		1.28
24DNT	C7H5N2O4	71	300†	1.442
Naphthalens	C ₁₀ H ₈	80,22	210.8 @ 720 torr	1.145
Acenaphthylene	C ₁₂ H8	92	265 - 275†	0.8988
Acenaphthene	C ₁₂ H ₁₀	96	278	1.225
Phenanthrene	C14H10	101	340	1.182
Anthracene	C ₁₄ H ₁₀	216.2	345	1.25
Fluoranthene	C ₁₆ H ₁₀	111	375	1.252
Pyrene	C ₁₆ H ₁₀	149	404	1.271
Chrysene	C18H10	254	448	1.274
Benzo(b)fluoranthene	C ₂₀ H ₁₂	167		
Benzo(k)fluoranthene	C ₂₀ H ₁₂	217		
Benzo(a)pyrene	C ₂₀ H ₁₂	179	312 @ 10 torr	
Dibenzo(a,h)anthracene	C22H14	269		
Indeno(1,2,3-cd)pyrene	C22H12	162.5	-	

^{*} There are four polymorphic structures of HMX with this range of densities.

[†] Decomposes.



injector and the guard column are maintained at room temperature. The silica precolumn and filter are maintained at the same temperature (52°C) as the analytical columns, which consist of an Ultrasphere CN 5-um column (25 cm by 4.6-mm ID) connected in series with an Ultrasphere ODS 5-um column (25 cm by 4.6-mm ID). The column temperature is maintained at 52°C by use of a circulating water bath (Fisher Scientific Model 80) and a column water jacket from Altech Associates. Any of the commonly available column thermostats capable of handling two columns may be substituted. The Ultrasphere CN column is the first column in the series after the injector followed by the Ultrasphere ODS column.

The column effluent is passed through three detectors in series, each connected by means of low-dead-volume unions and a minimum length of 0.010-inch stainless-steel tubing. The order of the detectors is as follows:

- 1. Perkin-Elmer LC-75 variable-wavelength spectrometer with autocontrol.
- 2. Altex Model 153 fixed-wavelength detector set at 254 nm.
- 3. Perkin-Elmer Fluorescence Spectrometer Model 650-S.

Each detector is connected to a Spectra Physics Model 4100 integrator. An Altex Model 420 microprocessor is used to control the pumps and signal the variable-wavelength detector. The microprocessor initially signals the autosampler to load the sample loop. The autosampler then flushes the sample loop for 60 seconds, injects the sample onto the analytical column, and signals the three integrators to start. After 10 minutes, the microprocessor sends a second flag to the autosampler to reset it prior to the next injection. At 60 minutes, the microprocessor signals the Perkin-Elmer LC-75 with autocontrol to switch detection wavelength from 230 to 280 nm. This wavelength

is reset to 230 nm at 135 minutes by another signal from the microprocessor, and at 137 minutes, a second signal to the LC-75 resets the detector for the next injection.

B. HPLC INSTRUMENTAL PARAMETERS

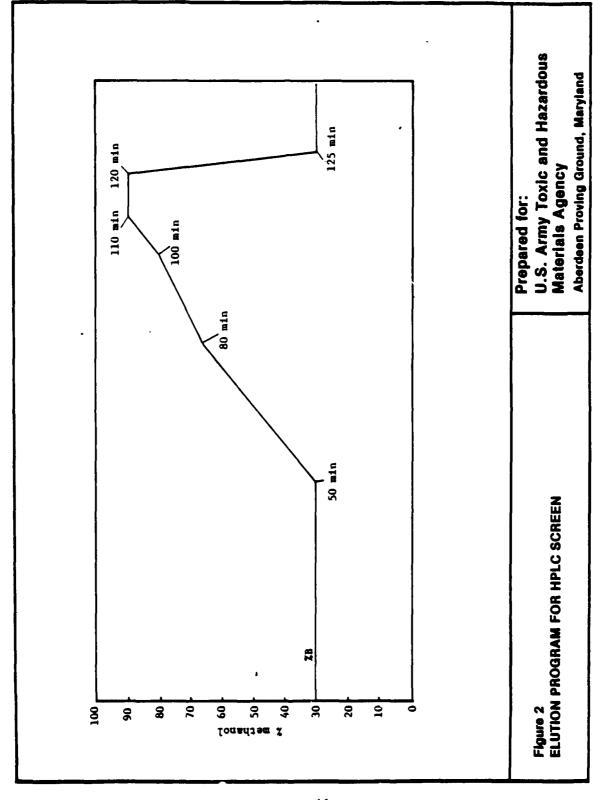
1. Detectors:

- a. Perkin-Elmer LC-75 variable-wavelength spectrometer with autocontrol set at 230 nm for first 60 minutes of chromatogram then switched to 280 nm.
- b. Altex Model 153 fixed-wavelength detector (254 nm).
- c. Perkin-Elmer 650-S fluorescence spectrometer.

 Excitation wavelength is 290 nm with a spectral band pass of 10 nm. Emission is monitored at wavelengths longer than 350 nm by use of a cutoff filter and by setting the emission monochromator in the zero-order mode.

2. Columns

- a. Guard column (5.0 cm by 4.6-mm) packed with Pelliguard LC-CN 40-um pellicular packing.
- b. Ultrasphere CN, 5-um column (25 cm by 4.6-mm ID).
- c. Ultrasphere ODS, 5-um column (25 cm by 4.6-mm ID).
- 3. Flow Rate: 1.0 ml/min.
- 4. Mobile Phase: Elution gradient (see Figure 2):
 - a. 30% methanol (CH₃OH) in phosphate-buffered water (pH=3) for 50 minutes.
 - Increase percentage of CH₃OH to 65% over
 30 minutes.
 - c. At 80 minutes, increase the percentage of CH₃OH to 80% over 20 minutes.
 - d. At 100 minutes, increase the percentage of CH₃OH to 90% over 10 minutes, and hold for 10 minutes.
 - e. Decrease the percentage of CH₃OH to 30% over 5 minutes, and hold for 15 minutes for reequilibration before next injection.



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- 5. Temperature: 52°C.
- 6. Injection Volume: 50 ul, fixed loop.
- 7. Retention Times (see Table 9).

C. HARDWARE/GLASSWARE

- 1. 2-liter separatory funnel (Teflon® or glass) (8).
- 2. 500-m1 K-D flask (8).
- 3. 20-ml K-D receiver (8).
- 4. Sep-Pak® silica-gel disposable cartridge (8) from Waters Associates.
- 5. 3-ball Snyder column (8).
- 6. 2-ball micro-Snyder column (8).
- 7. 10-ml graduated centrifuge tubes (8).
- 8. 10-ml syringe with Luer-lock fittings (1).
- 9. 4-cm glass funnels (8).
- 10. Disposable glass pipettes.

D. CHEMICALS

- 1. Nanograde methylene chloride--J.T. Baker Company.
- 2. Reagent-grade sodium chloride--J.T. Baker Company.
- 3. HPLC-grade methyl alcohol--J.T. Baker Company.
- 4. HPLC-grade water--J.T. Baker Company.
- 5. Anhydrous sodium sulfate--J.T. Baker Company.
- 6. 85-percent H₃PO₄, reagent-grade--J.T. Baker Company.
- 7. Teflon® boiling chips.
- 8. Colorphast® pH indicator sticks -- MCB Manufacturing Chemists, Inc.
- 9. 6N NaOH-240 g of reagent-grade NaOH pellets dissolved in 1 L of organic-free water.
- 10. 6N HC1--dilute-concentrated, reagent-grade HC1 in 1 L with organic-free water.
- 11. Sulfuric acid, reagent grade-J.T. Baker Company.
- 12. Sodium thiosulfate, reagent grade--J.T. Baker Company.

Table 9. HPLC Instrumental Parameters: Retention Times

Analyte	Retention Time (Minutes)
IMX	9.2
RDX	13.0
135TNB	16.8
13DNB	20.8
35DNP	26.9
246TNT	29.3
26DNT	35.0
24DNT	36.1
Naphthalene	84.5
Acenaphthylene	89.1
Acenaphthene	95.7
Phenanthrene	97.5
Anthracene	98.8
Fluoranthene	102.2
Pyrene	103.4
Chrysene	108.2
Benzo(b)fluoranthene	112.9
Benzo(k)fluoranthene	113.3
Benzo(a)pyrene	114.0
Dibenzo(a,h)anthracene	116.8
Indeno(1,2,3-cd)pyrene	117.9

4. STANDARDS

A. CALIBRATION STANDARDS

- Separate stock calibration standards are prepared for each analyte by weighing the indicated amounts of each compound into volumetric flasks and diluting to the mark with HPLC-grade acetonitrile (see Table 10).
- Intermediate stock calibration standards are prepared for some of the analytes by pipetting the indicated volumes of the stock calibration standards into volumetric flasks and diluting to the mark with HPLC-grade acetonitrile.

Analyte	Volume of Stock Calibration Standard Used (ml)	Final Volume (ml)	Concentration of Intermediate Stock Calibration Standard (ug/ml)
Phenanthrene	2	10	380
Chrysene	1	10	100
Benzo(b)fluoranthene	0.5	10	52
Benzo(k)fluoranthene	1	10	103
Benzo(a)pyrene	1	10	112
Dibenzo(a,h)anthracene	1	10	94
Indeno(1,2,3-cd)pyrene	. 1	100	36

3. The most concentrated (10x level) composite working calibration standard, F, is prepared from the separate stock and intermediate stock calibration standards. This high-level standard is diluted to yield all of the lower concentration standards. The high-level standard is prepared by pipetting the indicated amounts of the individual stock calibration standards or intermediate stock calibration standards into a single 50-ml volumetric flask. Fifteen milliliters of HPLC-grade acetonitrile are then added to make the final solution approximately 30% with

Table 10. Preparation of Calibration Standards

Analyte	Amount (mg)	Final Volume (ml)	Stock Calibration Standard Concentration (ug/ml)
нмх	10	10	1,000
RDX	10	10	1,000
135TNB	9.5	10	950
13DNB	11	10	1,100
35DNP	18.15	10	1,815
246TNT	10	10	1,000
26DNT	11.4	10	1,140
24DNT	10	10	1,000
Naphthalene	20.61	10	2,061
Acenaphthylene	32.8	10	3,280
Acenaphthene	12.0	10	1,200
Phenanthrene	19.0	10	1,900
Anthracene	30.3	100	303
Fluoranthene	10	10	1,000
Pyrene	9.6	10	960
Chrysene	10	10	1,000
Benzo(b)fluoranthene	10.4	10	1,040
Benzo(k)fluoranthene	10.3	10	1,030
Benzo(a)pyrene	11.2	10	1,120
Dibenzo(a,h)anthracene	9.4	10	940
Indeno(1,2,3-cd)pyrene	36	10	3,600

respect to acetonitrile. The solution is then diluted to the mark using phosphate-buffered, HPLC-grade water (pH = 3) (see Table 11).

4. The lower-level working calibration standards are prepared by pipetting the indicated volumes of the F standard into volumetric flasks and diluting to the mark with 30% acetonitrile in HPLC-grade water buffered with phosphate (pH = 3).

Standard	Volume of F Standard	Final Volume	Nominal Level
A	0	10	Blank
В	0.5	10	0.5x
С	1.0	10	1x
D	2.0	10	2 x
E	5.0	10	5 x

B. CONTROL SPIKES

 Intermediate stock spiking solutions are prepared for several of the heavier PAH compounds by pipetting the indicated volumes of the stock calibration standards into volumetric flasks and diluting to the mark with HPLC-grade acetonitrile.

Analyte	Volume of Stock Calibration Standard Used (ml)	Final Volume (ml)	Concentration of Intermediate Stock (ug/ml)
Phenanthrene	2	10	380
Chrysene	1	10	100
Benzo(b)fluoranthene	0.5	10	52
Benzo(k)fluoranthene	1.0	10	103
Benzo(a)pyrene	1.0	10	112
Indeno(1,2,3-cd)pyrene	1.0	100	36

Table 11. Preparation of the Most Concentrated Composite Working Calibration Standard, F

Analyte	Volume of Standard Diluted (ml)	Standard Diluted	Concentration of F Standard (ug/ml)
нмх	0.5	Stock	10
RDX	0.5	Stock	10
135TNB	0.5	Stock	9.5
13DNB	0.5	Stock	11
35DNP	0.5	Stock	18.2
246TNT	0.5	Stock	10
26DNT	0.5	Stock	11.4
24DNT	0.5	Stock	10
Naphthalene	0.5	Stock	20.6
Acenaphthylene	0.5	Stock	32.8
Acenaphthene	0.5	Stock	12
Fhenanthrene	1.0	Intermediate Stock	7.6
Anthracene	1.0	Stock	6.1
Fluoranthene	0.1	Stock	2.0
Pyrene	0.5	Stock	9.6
Chrysene	0.25	Intermediate Stock	0.5
Benzo(b)fluoranthene	0.5	Intermediate Stock	0.52
Benzo(k)fluoranthene	0.5	Intermediate Stock	1.03
Benzo(a)pyrene	0.5	Intermediate Stock	1.12
Dibenzo(a,h)anthracene	2.0	Intermediate Stock	3.76
Indeno(1,2,3-cd)pyrene	2.0	Intermediate Stock	1.44

- 2. The working control spike solutions are prepared by making a composite spike solution for the nitrosubstituted compounds and a separate solution for the PAHs. The indicated volumes (Table 12) of the stock calibration standards or intermediate stock spiking solutions are pipetted into a 25-ml volumetric flask and diluted to volume with HPLC-grade acetonitrile to make the working control spike solutions.
- 3. The following amounts of both the nitrocomposite and PAH composite working control spike solutions are pipetted into 1 L of standard or natural water. The concentrations spiked are shown in Section 8.

Nominal Level	Volume Spiked (ml)
Blank	0
0.5x	0.1
1x	0.2
2 x	0.4
5 x	1.0
10×	2.0

4. The precision, accuracy, and detection limits are determined for each analyte.

5. PROCEDURE

A. SAMPLING

- Samples must be collected in amber-glass containers with Teflon®-lined caps. The bottle must be prerinsed with the sample before collection.
- 2. Samples must be extracted within 7 days of collection.

Table 12. Preparation of Working Control Spike Solutions

	Volume		Final	
A - a 1 mb a	of Standard Diluted (m1)	Standard Diluted	Volume (ml)	Concentration
Anslyte	Diluced (ml)	Diluted	(mr)	(ug/ml)
Nitrosubstituted Compo	undsWorking (Control Spike	Solution	
HMX	0.5	Stock	25	20
RDX	0.5	Stock	25	20
135TNB	0.5	Stock	25	19
13DNB	0.5	Stock	25	22
35DNP	0.5	Stock	25	36.3
246TNT	0.5	Stock	25	20
26DNT	0.5	Stock	25	22.8
24DNT	0.5	Stock	25	20
PAH CompoundsWorking	Control Spike	Solution		
Naphthalene	0.5	Stock	25	41.2
Acenaphthylene	0.5	Stock	25	65.6
Acenaphthene	0.5	Stock	25	24
Phenanthrene	1	Intermediate	25	15.2
		Stock		
Anthracene	1	Stock	25	12.1
Fluoranthene	0.1	Stock	25	4.0
Pyrene	0.5	Stock	25	19.2
Chrysene	0.25	Intermediate Stock	25	1.0
Benzo(b)fluoranthene	0.5	Intermediate Stock	25	1.04
Benzo(k)fluoranthene	0.5	Intermediate Stock	25	2.06
Benzo(a)pyrene	0.5	Intermediate Stock	25	2.24
Dibenzo(a,h)anthracene	2	Intermediate Stock	25	7.52
Indeno(1,2,3-cd)pyrene	2	Stock Intermediate Stock	25	2.88

B. EXTRACTION

- Allow samples to warm to room temperature. Mark the water meniscus on the side of the sample container for later determination of the exact sample volume. The sample volume should not be less than 1 L. Do not filter the water.
- 2. Pour the entire sample into a 2-L glass or Teflon® separatory funnel with Teflon® stopcocks.
- 3. Check the pH with wide-range pH paper and adjust the pH to less than 3 with 6N HCl.
- 4. Add 100 g of reagent-grade NaCl, and shake to dissolve the salt.
- 5. Add 100 ml of methylene chloride to the sample bottle, shake for 30 seconds to rinse the container walls, and transfer the solvent into the separatory funnel.
- 6. Extract the sample by vigorously shaking the separatory funnel for at least 2 minutes with periodic venting to release any vapor pressure.
- 7. Allow the organic layer to separate from the water layer for a minimum of 10 minutes.
- 8. If the emulsion interface between the layers is greater than one-third the volume of the organic layer, centrifugation or placing the separatory funnel in an ultrasonic bath must be employed to break the emulsion. Addition of small amounts of methanol can also aid in dispersing emulsions.
- 9. Draw off the methylene chloride and pass through a glass funnel fitted with a small plug of glass wool and approximately 1 inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 25-ml K-D receiver, calibrated at the 25-ml mark.
- 10. Repeat Steps 5 through 9 two more times.
- 11. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride. This solvent rinse is added to the K-D apparatus.

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- 12. Add a Teflon® boiling chip to the methylene chloride extract in the flask, and attach a 3-ball Snyder column to the apparatus. Prewet the Snyder column by adding approximately 1 ml of methylene chloride to the top.
- 13. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
- 14. The balls of the Snyder column should actively chatter, but the chambers should not flood.
- 15. When the apparent volume of the liquid in the receiver is less than approximately 15 ml, remove the K-D apparatus from the water bath and allow it to drain at least 10 minutes while cooling.
- 16. Remove the Snyder column and rinse the K-D flask and its lower joint into the receiver with 1 to 2 ml of methylene chloride.
- 17. Raise the volume of the methylene chloride in the receiver to the calibrated 20-ml mark.
- 18. Stopper the receiver with an ungreased ground-glass stopper, and mix the extract by inversion of the receiver.
- 19. Remove the ground-glass stopper, and pipette 10 ml of the methylene chloride extract into an amber or foil-wrapped 20-ml glass vial. Cap the vial with a Teflon®-lined cap, and store at 4°C for later use if a silica-gel cleanup is necessary.
- 20. Rinse the ground-glass stopper with 1 to 2 ml of methylene chloride into the extract remaining in the receiver.
- 21. Place a micro-Snyder column on the receiver, and prewet the column with 1 ml of methylene chloride. Add a fresh Teflon boiling chip.
- 22. Concentrate the extract by gently heating the receiver in an 80°C water bath.

- 23. When the apparent volume of the liquid is less than approximately 1 ml, remove the receiver from the water bath.
- 24. Remove the micro-Snyder column and rinse its lower joint into the receiver with 2 ml of HPLC-grade acetonitrile.
- 25. Reattach the 2-ball micro-Snyder column, and reconcentrate to 0.5 ml.
- 26. Remove the receiver from the water bath and again add 2 ml of HPLC-grade acetonitrile, rinsing the joint column with the solvent.
- 27. Again, reconcentrate the sample to 0.5 ml.
- 28. Repeat Steps 26 and 27.
- 29. After this third exchange to acetonitrile, the extract is quantitatively transferred to a 10-ml graduated centrifuge tube.
- 30. The extract volume is then reduced by controlled evaporation under a gent'e stream of dry nitrogen to a volume of approximately 0.2 to 0.3 ml.
- 31. The sides of the graduated centrifuge tube are then rinsed with 0.2 ml of HPLC-grade acetonitrile to yield an approximately 0.6-ml extract volume.
- 32. Dilute the extract to the calibrated 2-ml mark with phosphate-buffered, HPLC-grade water (pH = 3). This procedure yields a final extract volume of 2 ml in a solution that contains approximately 30% CH₃CN.
- 33. Transfer this extract to a 1-ml septum-sealed vial for HPLC analysis.
- 34. The extract is now ready for HPLC analysis.

C. SAMPLE CLEANUP

If it becomes apparent during the HPLC analysis that the sample cleanup is necessary for proper qualitative identification of the sample components, the following procedure is used to help remove possible interferents. The cleanup procedure is necessary when components are found with the proper retention times but do not have the correct detector ratios, or when large broad-band interferents are noted in the chromatogram. The sample cleanup steps are as follows:

- 1. The 10-ml extract portion saved in Step 19 of the extraction procedure is transferred to a 10-ml glass syringe with Leur-lock tip.
- 2. The extract is then passed through a silica-gel Sep-Pak® attached to the syringe at a rate of approximately 5 ml/min, and the eluate is collected in a 20-ml K-D receiver.
- 3. The storage vial is rinsed with 5 ml of 50% CH₃OH in methylene chloride; this extract is passed through the silica-gel Sep-Pak® and the eluate collected.
- 4. The eluates from Steps 2 and 3 may be combined if only general cleanup is desired. The eluates are analyzed separately if sample fractionation is also required.
- 5. Follow Steps 21 through 34 of the extraction procedure to prepare the final extract for HPLC analysis.

D. CALIBRATION

- A minimum of three instrument calibration standards and a blank will be run at the beginning of the analytical run.
 One of these standards will be duplicated at the conclusion of the analytical run to verify constant instrument response.
- 2. Plot the normalized integrator areas versus micrograms/ milliliter of each standard to obtain a working curve.

E. ANALYSIS

- 1. Inject 50 ul of the extract onto the HPLC column.
- 2. Perform the analysis of the sample according to the conditions given in Section 3(B).
- 3. Measure the response of the sample for the components of interest on each of the three detectors. Peak heights

rather than areas are used because they are less subject to interferences.

- 4. Quantitation of the components of interest is carried out according to Section 6 on the particular detector specified for each analyte in Section 1.
- 5. Qualitative identification of the component of interest is outlined in Figure 3. Detector ratios are calculated for unknown sample components and compared to the ratios obtained during the calibration run. The ratios obtained during documentation should serve as guidelines for the magnitude and variance of the expected ratios. The absolute value of the ratios may vary somewhat on different instrumentation, especially on ratios involving fluorescence because only a relative intensity on a particular instrument is measured. The absorbance ratios should be more consistent among different instruments because the absorbance is a property measured in a more rigorously defined manner than fluorescent intensity.

If the retention time and the ratios match those for one of the standards, the presence of that particular analyte in the sample is confirmed. If peaks are found for which the retention times match but the ratios do not match any ci the standards, sample cleanup is conducted. The cleaned fraction is then analyzed by HPLC, and the same criteria are applied for compound verification.

A detector ratio is considered positive if it falls within the 95-percent confidence interval for the ratio obtained during documentation for that particular analyte. The mean detector ratios, 95-percent confidence interval, and lowest level (ppb) at which these ratios could be accurately measured due to instrumental sensitivity limitations are presented in Tables 13 and 14.

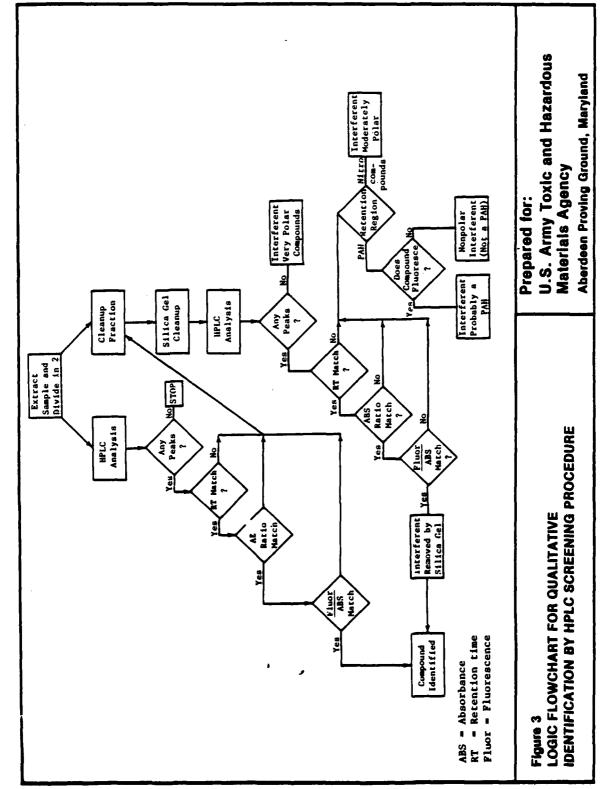


Table 13. Absorbance Ratios and Detection Limits of Analytes in Standard Water without Silics-Gel Clearup

Compound	Detector Wavelength (rm)	Mean Absorbance Ratio* + 95% CI†	Lowest Concentration for Ratio (ug/L)	Target IL (ug/L)	Documented Di.** (ug/L)	Retention Time (minutes)
XOEL	730754	9	0 4		, , ,	6
RDK	230/254	3.9 - 0.25	2.0	, c	j 4	7.2
ATHEA	230/254	2,8		£ 9	? ≨	14.3
135 TNB	230/254	ी । ।	1.9	3.8	ه و	16.8
1.3DNB	230/254	1.5 + 0.26	2.2	4.4	•	20.8
2	230/254	R.0	2	2	2	24.8
SONE	230/254	1.7 ± 0.58	3.6	7.3	11	26.9
Tetryl	230/254	3.14	2	2	2	29.5
246DNT	230/254	2.3 ± 0.43	2.0	4.0	9	25.3
26DNT	230/254	1.5 ± 0.95	2.3	4.6	6	35.0
24DNT	230/254	1.2 ± 0.31	2.0	4.0	9	36.1
1 20NT	230/254	0.852	2	2	2	52.5
Naphthalene	280/254	1.6 ± 0.8	4.1	8.2	72	84.5
	254/F1****	21 + 12	41	8.2	1	ł
	280/F1	36 ± 5.0	41	8.2	1	I
Acenephthylene	280/254	1.3 ± 0.28	6.6	13	77	89.1
Acenaphthene	280/254	0.53 ± 0.13	1.5	4. 8	10	95.7
Prematurene	280/254	0.35 ± 0.13	1.5	3.0	2	97.5
	254/FI	29 + 5.9	6.1	3.0	ı	1
	280/FI	11 + 1.6	6.1	3.0	i	1

Table 13. Absorbance Ratios and Detection Limits of Analytes in Standard Water without Silica-Gel Cleanup (Continued, Page 2 of 3)

Compound	Detector Wavelength (mm)	Absorbance Ratio* + 95% CI†	Lowest Concentration for Ratio (ug/L)	Target DL (ug/L)	Documented III# (ug/L)	Retention Time (minutes)
Anthracene	280/254	0.0082 ± 0.014	12	2.4	2	98.8
	254/FI	220 + 56	12	2.4	l	{
	280/FI	1.5 ± 0.31	12	2.4	t	1
Fluoranthene	280/254	2.2 ± 0.64	0.4	8.0	6.0	102.2
	254/FI	0.55 ± 0.16	0.4	9.0	ı	1
	280/FI	1.2 ± 0.39	. 6.0	8.0	ι	1
Pyrene	280/254	0.46 ± 0.16	1.9	3.8	œ	103.4
	254/FI	7.9 ± 1.5	1.9	3.8	l	1
	280/F1	3.8 ± 1.0	. 61	3.8	l	1
Chrysene	280/254	0.43 ± 0.15	0.40	07.0	7.0	108.2
	254/FI	15 + 1.3	1.0	0.20	ı	1
	280/FI	6.6 ± 2.5	1.0	0.20	1	1
Benzo(b)fluoranthene	254/280	0.99 ± 0.42	0.21	0.21	2	112.9
	254/FI	0.97 ± 0.27	0.21	0.21	i	1
	280/F1	0.99 ± 0.56	0.21	0.21	1	1
Benzo(k)fluoranthene	280/254	1.3 ± 0.47	0.21	0.41	-	113.3
	254/FI	0.39 ± 0.16	0.21	0.41	į	1
	280/F1	0.50 ± 0.23	0.21	0.41	ł	1
Benzo(a)pyrene	280/254	1.6 ± 0.70	0.45	0.45	9.0	114.0
	254/FI	1.3 ± 0.58	0.45	0.45	1	1
	280/FI	2.0 + 0.83	0.45	0.45	1	١

Table 13. Absorbance Ratios and Detection Limits of Analytes in Standard Water without Silica-Gel Cleanup (Ontinued, Page 3 of 3)

Compound	Detector Wavelength (m)	Hean Absorbance Ratio* + 95% CIT	Lowest Concentration for Ratio (ug/L)	Target DL (ug/L)	Documented LIL*** (ug/L)	Retention Tine (minutes)
Dibenzo(a,h)anthracene	280/254	12 ± 6.4	0.75	1.5	Q	116.8
	254/81	0.62 ± 0.39	0.75	1.5	1	ı
	280/FI	6.9 ± 2.2	0.75	1.5	ì	ţ
Indexo(1,2,3-cd)pyrene	280/254	0.89 ± 0.27	0.58	0.58	7 .	117.9
	254/FI	1.6 + 0.34	0.58	0.58	1	ı
	280/FI	1.4 ± 0.32	0.58	0.58	ı	1

* Determined from peak height measurements.

† CI = Confidence interval (n = 20).

*** DL = Detection limit calculated according to Hubans and Vos, 1970.

*** The determined due to co-elution, interference, or instability problems.

**** Fluorescence measured at wavelengths greater than 350 nm.

Table 14. Absorbance Ratios and Detection Limits of Analytes in Standard Water with Silica-Gel Cleanup

Compound	Detector Wavelength (rm)	Mean Absorbance Ratio* + 95% CI†	Lowest Concentration for Ratio (ug/L)	Target DL (ug/L)	Documented II,44 (ug/L)	Retention Time (minutes)
BC	230/254	6.6 + 2.2	4.0	4.0	17	9.2
XOX	230/254	3.0 ± 0.63	2.0	4.0	80	13.0
135mB	230/254	3.6 + 0.79	1.9	3.8	9	16.8
13DB	230/254	1.9 ± 0.29	2.2	4.4	80	20.8
35CNP	230/254	1.6 ± 0.68	3.6	7.3	œ	26.9
246INI	230/254	2.3 ± 0.76	2.0	4.0	11	29.3
26DNT	230/254	1.7 ± 0.89	2.3	4.6	21	35.0
24DNT	230/254	1.1 ± 0.55	2.0	4.0	7	36.1
Naphthalene	280/254	1.6 ± 0.62	4.1	8.2	83	84.5
	254/1111	22 + 16	41	8.2	1	ı
	280/F1	37 + 6.0	17	8.2	1	ı
Acenephthy lene	280/254	1.4 + 0.71	9.9	13	33	89.1
Acenaphthene	280/254	0.52 ± 0.47	1.5	4.8	71	95.7
Phenanthrene	280/254	0.35 + 0.05	1.5	3.0	٠,	97.5
	254/FI	31 + 12	6.1	3.0	i	ļ
	280/FL	10 + 4.7	6.1	3.0	ı	ļ
Anthracene	280/254	0.0084 + 0.011	71	2.4	5	8.8
	254/FI	20 + 72	71	2.4	ı	ı
	280/F1	1.9 ± 2.5	71	2.4	1	ı

Table 14. Absorbance Ratios and Detection Limits of Analytes in Standard Water with Silica-Gel Cleanup (Page 2 of 3)

Compound	Detector Wavelength (rm)	Mean Absorbance Ratio* + 95% CI†	Lowest Concentration for Ratio (ug/L)	Target IL (ug/L)	Documented IL** (ug/L)	Retention Time (minutes)
The same of the sa	280/256	340	70	8	-	681
	700 /m		•		•	102:1
	254/FI	0.53 + 0.13	7.0	8.0	١	ı
	280/F1	1.2 ± 0.30	7.0	9.0	1	i
Pyrene	280/254	0.48 ± 0.17	1.9	3.8	9	103.4
	254/FI	7.8 + 1.4	7.7	3.8	1	1
	280/FI	3.7 ± 0.97	7.7	3.8	l	ı
Chrysene	280/254	0.40 + 0.046	07.0	0.20	0.5	108.2
	254/FI	16.2 ± 5.9	1.0	0.20	l	l
	280/FI	6.0 ± 5.0	1.0	0.20	I	ı
Benzo(b) fluoranthene	280/254	1.0 ± 0.46	0.21	0.21	0.3	112.9
	254/FI	0.94 + 0.30	0.21	0.21	ı	ı
	280/F1	0.96 + 0.38	0.21	0.21	ı	ļ
Benzo(k)fluoranthene	280/254	1.4 ± 0.71	0.41	0.41	0.8	113.3
	254/FI	0.39 ± 0.26	0.21	0.41	ı	ı
	280/F1	0.53 ± 0.26	0.41	0.41	ļ	ı
Benzo(a)pyrene	280/254	1.8 ± 0.52	0.45	0.45	0.9	114.0
	254/FI	1.3 ± 0.51	0.45	0.45	ţ	1
	280/FI	2.0 ± 1.2	0.45	0.45	ŧ	ı

Table 14. Absorbance Ratios and Detection Limits of Analytes in Standard Water with Silica-Gel Clearup (Page 3 of 3)

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Compound	Detector Wavelength (mm)	kean Absorbance Ratio* + 95% C.I.†	Lowest Concentration for Ratio (ug/L)	Target DL (ug/L)	Document ed IL*** (ug/L)	Retention Time (minutes)	
Dibenso(a,h) anthracene	280/254	13 ± 3.7	1,5	1.5	Wester	116.8	ł
	254/FI	0.61 ± 0.28	1.5	1.5	1	ı	
	280/F1	7.7 ± 2.9	1.5	1.5	ı	ı	
Indeno(1,2,3-cd)pyrene	280/254	0.88 ± 0.38	0.58	0.58	2	117.9	
	254/FI	1.6 ± 0.77	0.58	0.58	1	I	
	280/F1	1.4 ± 1.0	0.58	0.58	I	i	

* Determined from peak heights measurements.

† CI = Confidence interval (n = 20).

Source: ESE, 1962.

6. CALCULATIONS

Determine the concentration of each analyte according to the following formula:

Concentration (ug/g) = $\underline{(A)(V_t)}$

V.

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve (ug/ml),

 $V_t = Volume of total extract (m1), and$

 V_s = Volume of initial sample extracted (L).

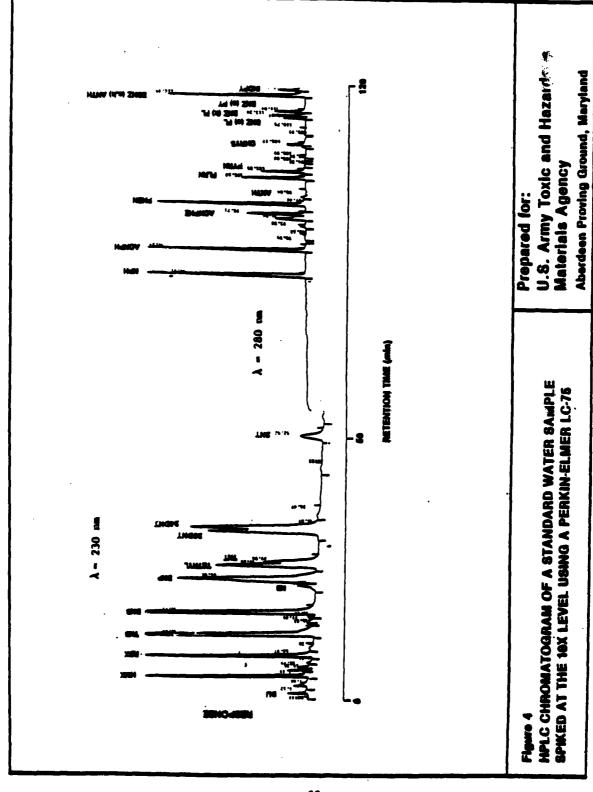
The concentration is corrected for recovery by dividing by the slope of the regression line for observed value versus target value for spiled samples.

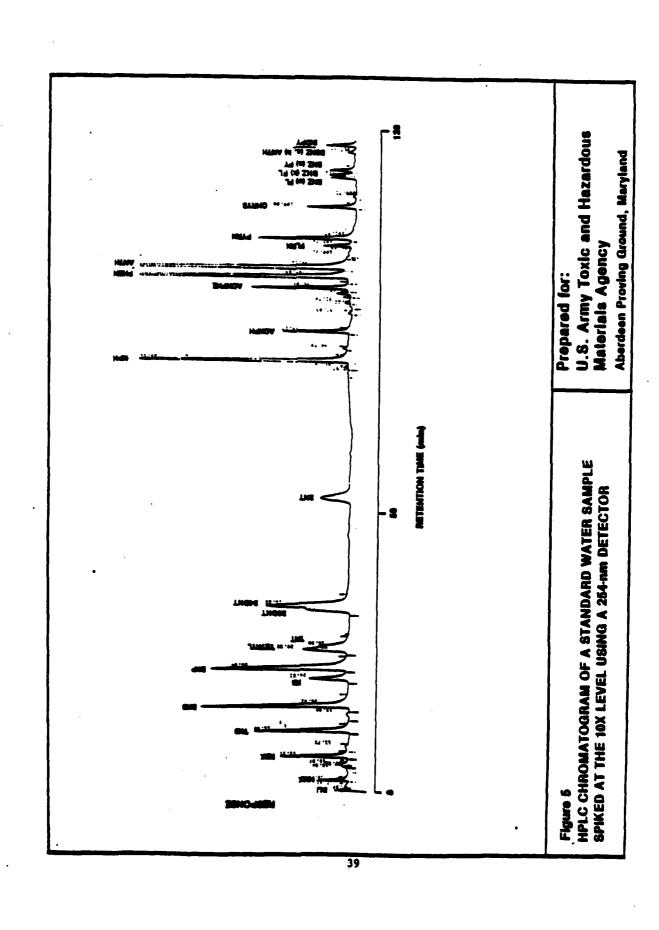
7. REFERENCES

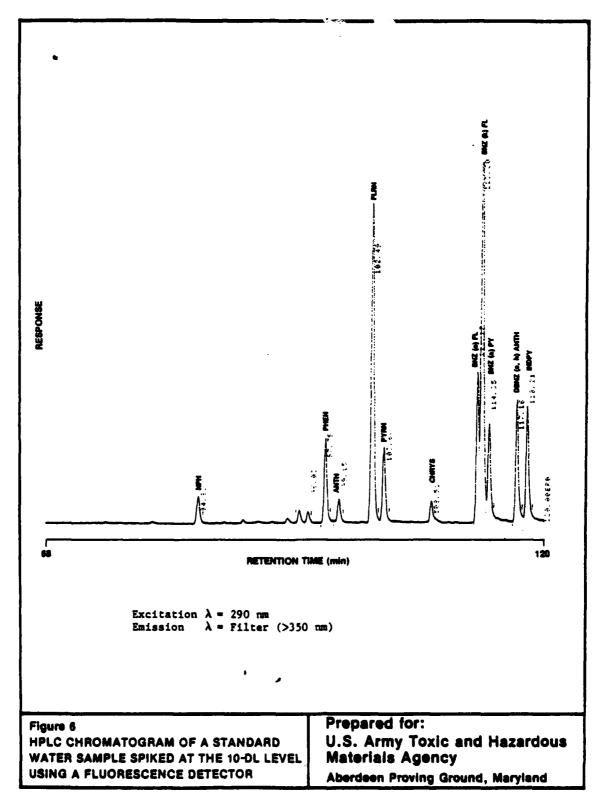
Hubaux, A. and Vos, G. 1970. Anal. Chem. 42, 849-885.

8. DATA

See Figures 4 through 6 and attached data sheets.

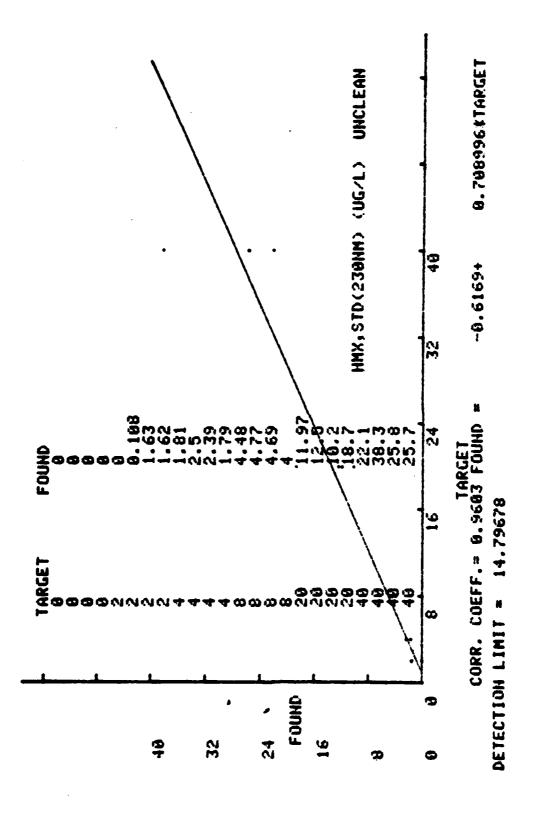






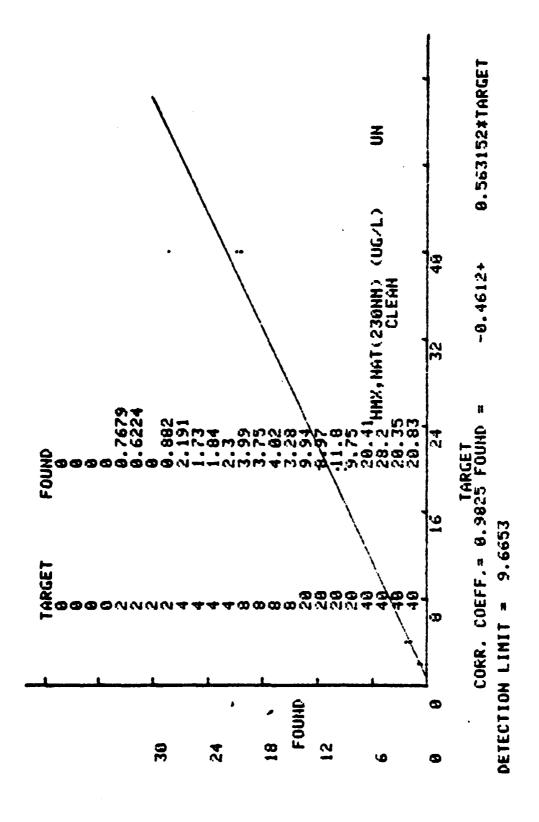
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0.0000	0.0000	0.0000	0.0000	0.0000	
2.00	0.0000	0.108	1.63	1.62	
4.60	1.81	2.50	2.39	1.79	
8 • 7 0	4.48	4.77	4.69	4.00	
20.0	12.0	12.5	10.2	18.7	
40.0	22.1	38.3	25.8	25.7	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.000	0.5000
2.00	0 ∙839	0.908	108	-58. 9
4 • 0 0	2.12	0.375	17.7	-46.9
8 • 0 0	4.48	0.346	7.71	-43 .9
20.0	13.3	3.70	27.8	-33.3
40.0	28.0	7.18	25.4	-30.1



HMX, NAT (230NM) (U	IG/L) UNCL	EAN		•	
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.000	0.0000	0.0000	
2.00	0.768	0.622	0.0000	0.882	
4.00	2.19	1.73	1.84	2.30	
20.8	3.99	3.75	4.02	3.28	,
20.0	9.94	8.97	11.8	9.75	
40.0	20.4	28.2	20.3	20.8	

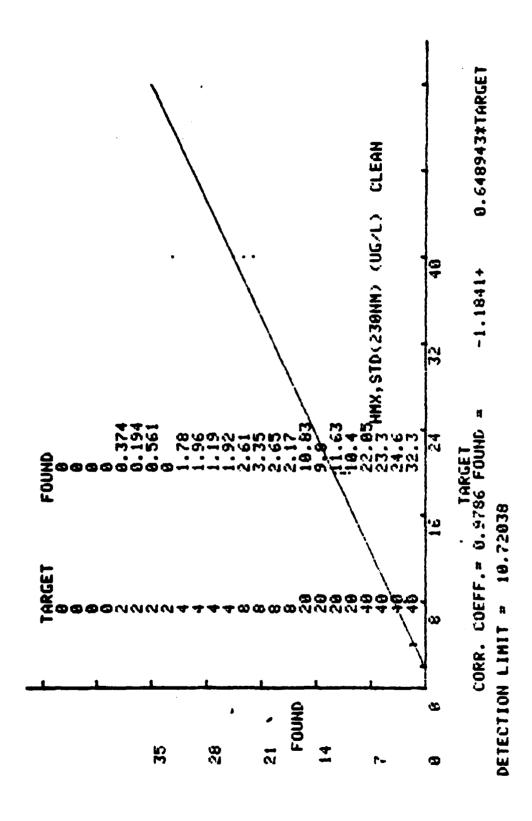
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.00	0.568	0.393	69.2	-71.6
4.00	2.02	0.273	13.6	-49.6
8.00	3.76	0.342	9.10	-53.0
20.0	10.1	1.20	11.9	-49.4
40 • C	22.4	3.84	17.1	-43.9



HMX.STD(230NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.8020	0 • 0 0 0 C	1.0000	
2.09	0.374	9.194	0.561	9.0000	
4.00	1.78	1.96	1.19	1.92	
8 • 0 0	2.61	3.35	2.65	2.17	
20.0	10.8	9.80	11.6	10.4	
40.0	22.0	23.3	24.6	32.3	

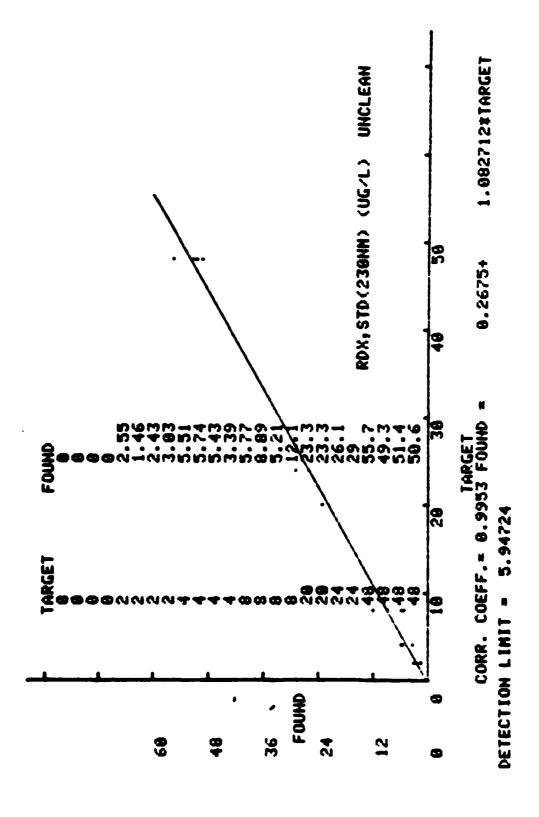
TARGET CONCENTRATION	AVERAGE Found value	STANDARD DEVIATION	PERCENT Imprecision	PERCENT INACCURACY	
0.0000	0.000	0.0000	0.0000	0.9673	
2.00	0 •282	0.241	85.2	-85. 9	
4 • 0 0	1.71	0.357	20.8	-57.2	
8.00	2.69	0.488	18.1	-66.3	
20.0	10.7	0.770	7.22	-46.7	
40.5	25.6	4.61	18.0	-36.1	



RDX.STD(230NM) (UG/L) UNCLEAN

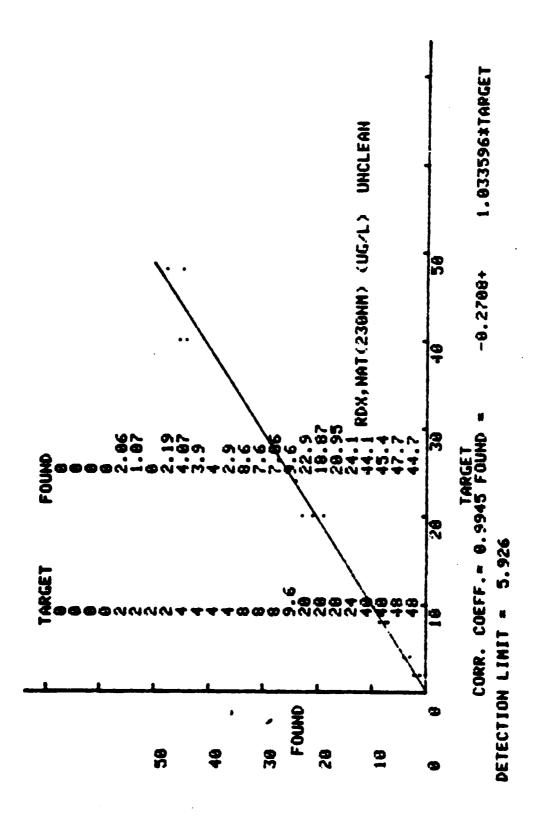
TARGET CONCENTRATION	1	DAY 2	3	4	
0.000	0.0000	0.0000	0.0000	0.0000	
2.00	2.55	1.46	2.43	3.03	
4.00	5.51	5.74	5.43	3.39	
8.30	5.77	8.89	5.21	12.1	
20.0	23.3	23.3	26.1	29.0	
48.0	55.7	49.3	51.4	50.6	-45

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT Imprecision	PERCENT INACCURACY
0.000	0.0000	0.0000	0.0008	0.0000
2.00	2.37	0.658	27.8	18.4
4.00	5.02	1.09	21.8	25•4
8.00	7.99	3.18	39.8	-0.0938
20.0	46 • 6	0.0000	0.0000	33.0
48.0	55 • 1	0.0000	0.0000	29.6



RDX NAT (230NM) (UG/	L) UNCLE	AN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0003	0.0000	0.0000	1.0000	
2.00	2.06	1.07	0.0000	2.19	
4 • 0 C	4.07	3.90	4.00	2.90	
8.00	8.60	7.60	7.06	9.60	
20.0	22.9	18.9	20.9	24.1	
40.0	44.1	45.4	47.7	44.7	

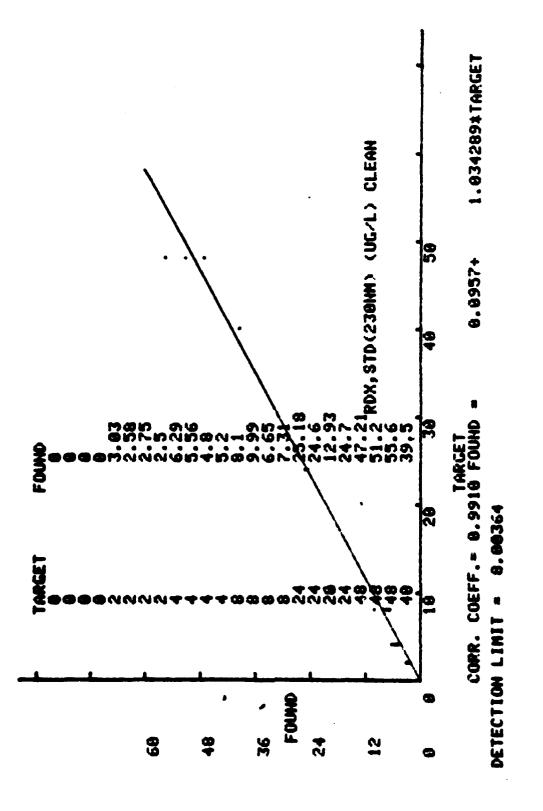
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.000	0.0000	0.0000	0.0000	0.0000
2.03	1.33	1.02	76.5	-33.5
4.08	3.72	0.549	14.8	-7.06
8.00	7.75	0.781	10.1	-3∘ ûα
20.0	9.60	0.0000	0.2000	C.c.u.c.o
40.0	20.9	2.02	9.64	4.53



ROX, STD (230NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	DAY	3	4	
0.0000	0.0000	0.0000	0.0000	0.000	
2.00	3.03	2.58	2.75	2.50	
4.50	6.29	5.56	4 - 80	5.20	
8.00	8-10	9.99	6•65	7.31	
20.0	12.9	25 • 2	24.6	24.7	
40.0	39.5	47.2	51.2	55.6	

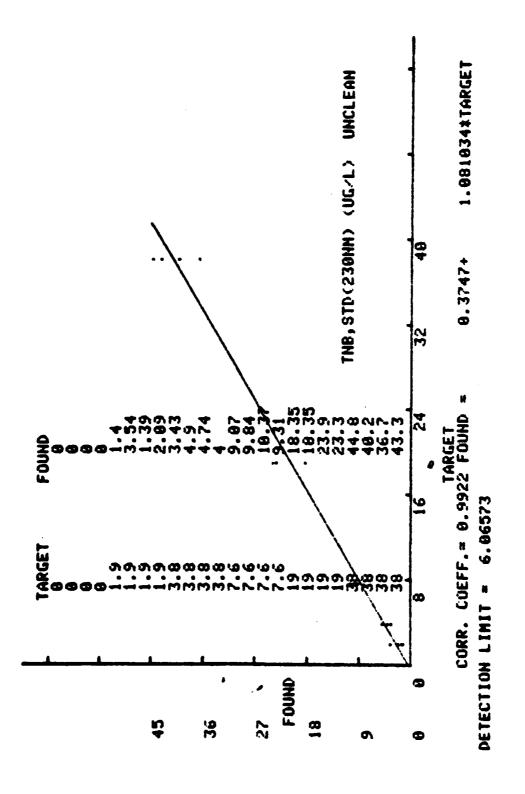
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT Imprecision	PERCENT INACCURACY
0.0000	0000	0.000	0.0000	0.000
2.00	2.71	0.234	8 • 6 4	35.7
4.30	5.46	0.633	11.6	36.6
8.00	8.01	1.45	18.0	0.156
20.0	12.9	0.0000	0.0000	-35.4
40.0	24.8	0.311	1.25	3.44



TNB.STD(230NM) (UG/L) UNCLEAN

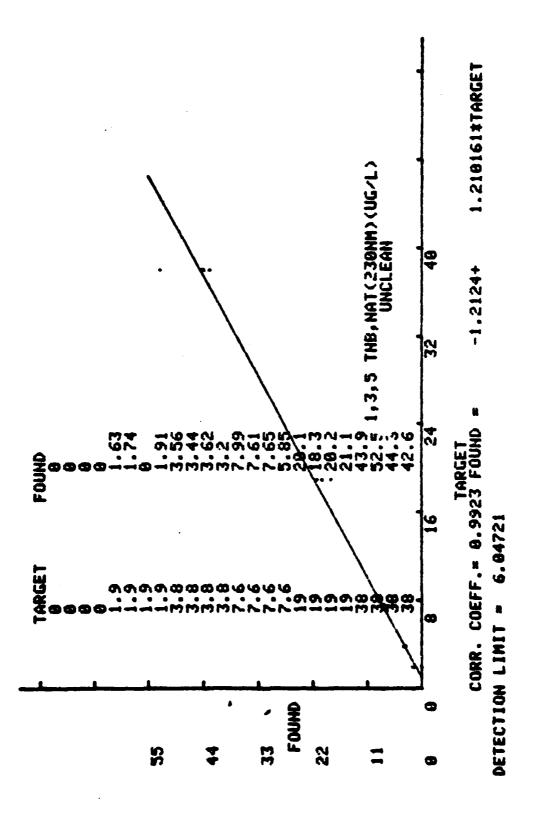
TARGET CONCENTRATION	1	DAY 2	3	4	
0.000	0.0000	0.0000	0.0000	0.0000	
1.90	1 • 4 0	3.54	1.39	2.09	
3.80	3.43	4.90	4.74	4 - 0 0	
7.60	9.07	9.84	10.4	9.31	•
19.0	18.3	18.3	23.9	23.3	
38.0	44.8	40.2	36.7	43.3	

TARGET CONCENTRATION	AVFRAGE Found Value	STANDARD DEVIATION	PERCENT Imprecision	PERCENT INACCUPACY	
0.0000	0.0000	0.0000	0.0000	0.000	
1.90	2.10	1.01	48.0	10.8	
3.80	4.27	0.682	16.0	12.3	
7.60	9.65	0.579	6.00	26.9	
19.0	21.0	3.04	14.5	10-4	
38.0	41.2	3.59	8.70	8.55	



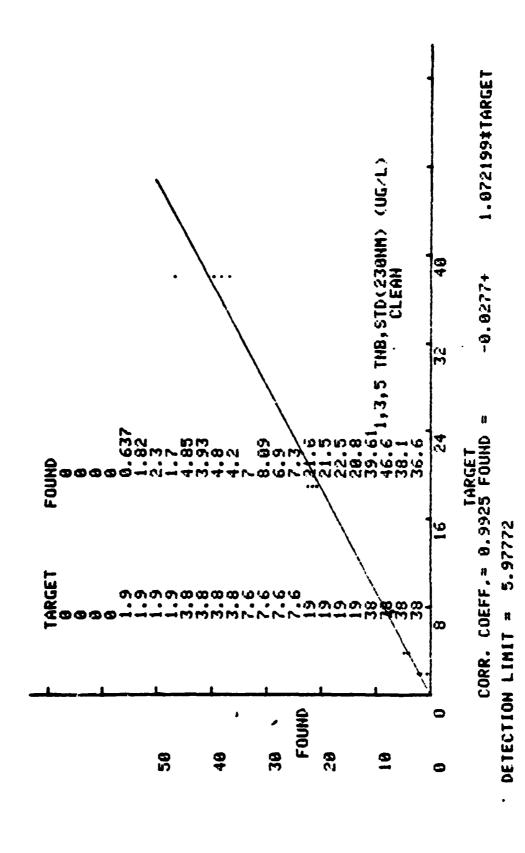
1.3.5 TNB.NAT(230NM)(UG/L)		UNCLEAR			
TARGET CONCENTRATION	:	DAY 2	3	4	
8.0 005	0.0000	0.0566	0.0909	0.0000	
1.50	1.63	1.74	0.0000	1.01	
3.00	3.56	3.44	3.62	3.20	
7.60	7.99	7.61	7.65	5.85	
19.0	20.1	18.3	20.2	21.1	
38.0	43.9	52.5	44.3	42.6	

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	: • 0 0 0 0	0.000	0.000	0.0000	
1.90	1.32	1.888	67.2	- 3°•5	
3.26	3.45	0.186	5.38	-9 -08	
7.6	7.27	0.965	13.3	-4. ?€	
19.0	19.9	1.17	5.69	4.87	
38.0	45.8	4.51	9 • 2 4	20•4	



1.3.5 TNB.STD(23SNM) (UG/L) CLEAN						
TARGET CONCENTRATION	1	DAY 2	3	4		
8.0600	0.0000	0.0000	0.2000	0.000		
1.90	0.637	1.82	2.30	1.70		
3.80	4.85	3.93	4.87	4 • 2 0		
7.60	7.00	8.09	6.90	7.39		
19.0	21.6	21.5	22.5	20.8		
38.0	39.6	46.6	38.1	36.6		

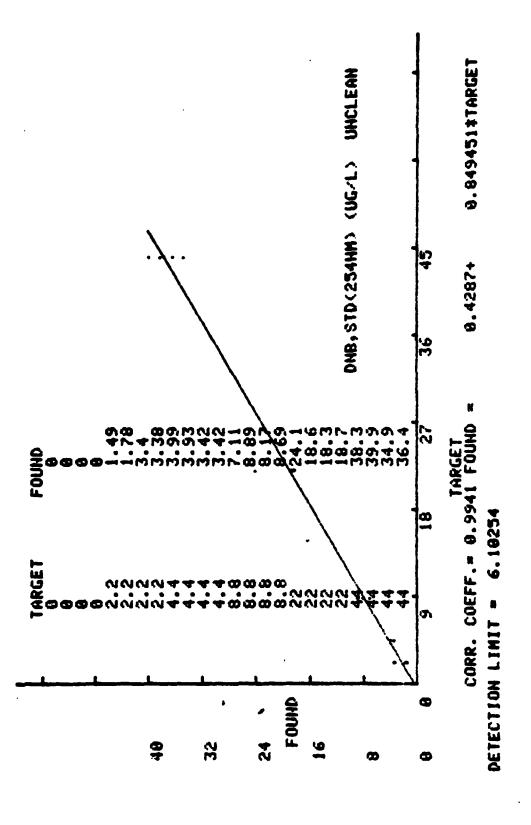
TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT Imprecision	PERCENT INACCURACY
8.0000	S • C O G C	0.0000	0.0000	0.9000
1.50	1.61	0.701	43.4	-15.9
3.80	4.44	0.453	10.2	17.
7.60	7.32	0.539	7.36	-3.6 =
19.0	21.6	0.698	3.23	13.7
38.0	40.2	4.42	11.0	5.86



DNB,STD(254NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0-0000	9.0000	
2.20	1.49	1.78	3.40	3.38	
4.40	3.99	3 • 9 3	3.42	3.42	
8.80	7-11	8.89	8.17	8.69	
22.0	24.1	18.6	18.3	18.7	•
44.0	38.3	39.9	34.9	36.4	

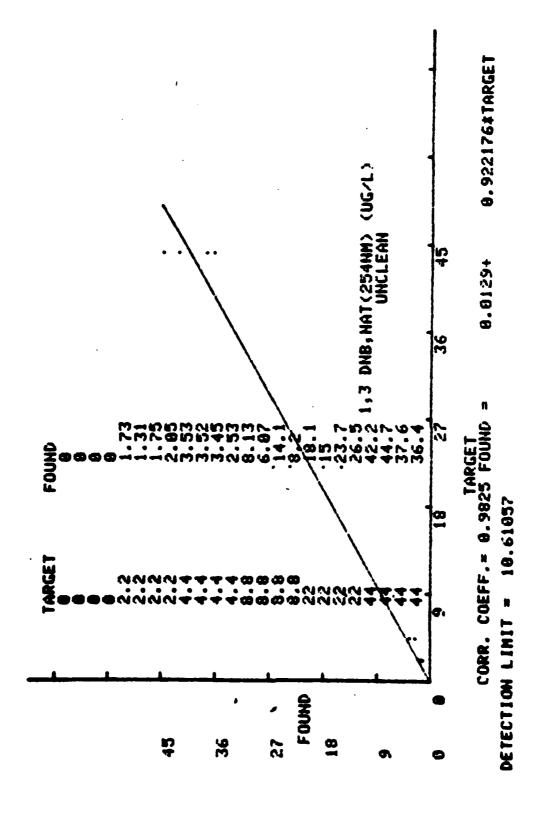
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.6000	0.000	0.0000	0.0000
2 • 2 0	2.51	1.02	40.6	14.2
4.40	3.69	0.313	8 • 48	-16.1
8.80	8.21	0.797	9.70	- 6 •65
22.0	19.9	2.79	14.0	-9.43
44.0	37.4	2.18	5.84	-15.1



1.3 DNB.NAT(254NM) (UG/L) UNCLEAN

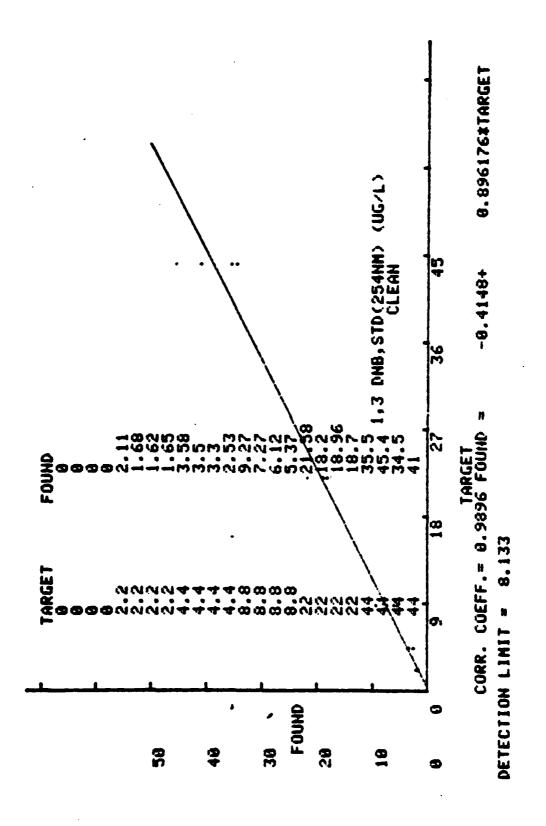
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	C.00C0	
2.20	1.73	1.31	1.75	2.05	
4.40	3.53	3.52	3.45	2.53	:
8.80	8.13	6.07	14.1	8.20	
22.3	18.1	15.0	23.7	26.5	
44.0	42.2	44.7	37.6	36.4	

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.9003	0.0000	0.000	0.000	0.0000	
2.20	1.71	0.364	17.8	-22.3	
4 • 4 û	3.26	0.486	14.9	-26.0	
8.80	9.12	3.46	37.9	3•69	
22.5	20.8	5.22	25.1	-5.34	
44.0	40.2	3.89	9.68	-8.58	



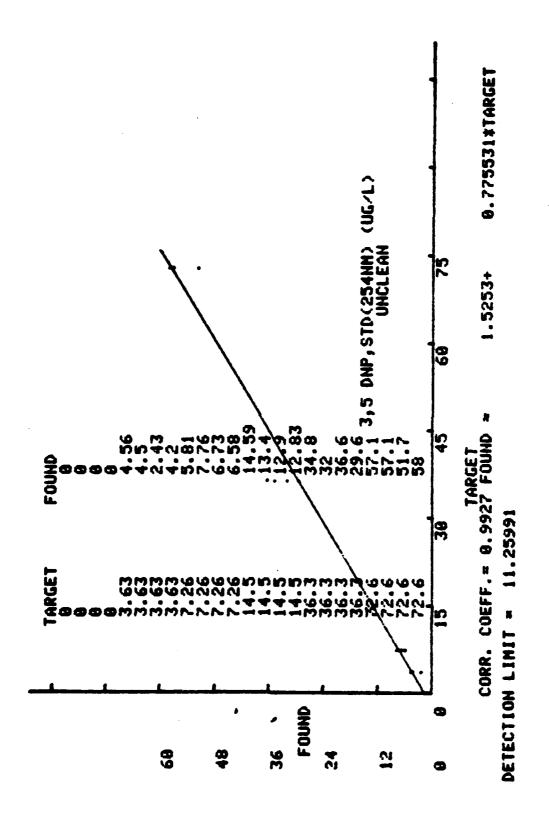
1.3 DNB.STD(254NM)	(U6/L)	CLEAN			
TARGET	•	DAY	3	4	
CONCENTRATION	1	2			
0.0000	0.0000	0.0000	0.0000	0.0000	
2.20	2.11	1.68	1.62	1.65	•
4.40	3.58	3.50	3.30	2.53	
8.86	9.27	7.27	6.12	5.37	
22.0	21.6	18.2	19.0	18.7	
44 • ū	35.5	45.4	34.5	41.0	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	9.0000	0 • 6 0 0 0	.0.0000	0.000	
2.20	1.76	0.231	13.1	-19.8	
4.40	3.23	9.486	14.9	-26.6	
8.80	7.01	1.70	24 • 2	-20.4	
22.0	19•4	1.51	7.82	-12.0	
44.0	39.1	5.08	13.0	-11.1	



3,5 DNP,STD(254NM)	(UG/L)	UNCLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	0.0000	
3.63	4.56	4.50	2.43	4.20	
7.26	5.81	7.76	6.73	6.58	
14.5	14.6	13.4	12.9	12.8	
36.3	34.8	32.0	36.6	29.6	
72.6	57.1	57.1	51.7	58.0	

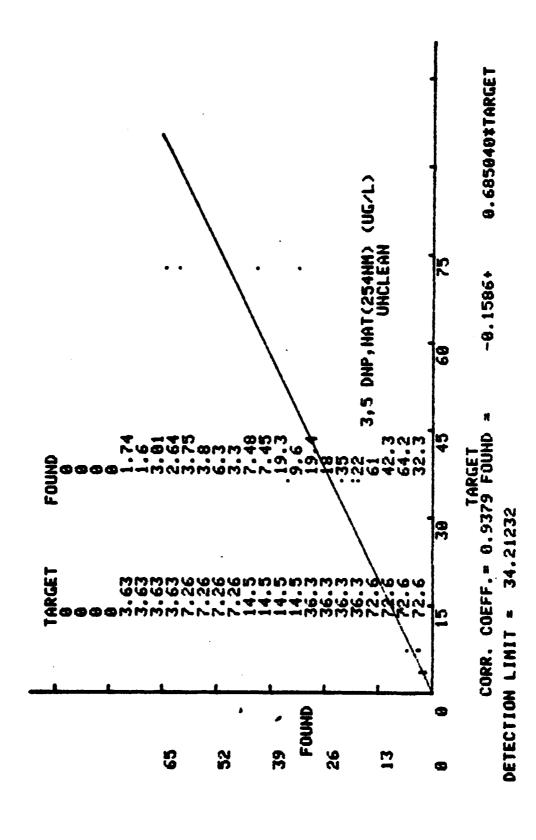
TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT Imprecision	PERCENT Inaccuracy	-
0.0000	0.0000	0.0000	0.0000	0.0000	
3.63	3.92	1.01	25.7	8.06	
7.26	6.72	0.802	11.9	-7.44	
14.5	13.4	0-814	6.06	-7.38	
36.3	33.2	3.08	9.27	-8 • 4 0	
72.6	56 • C	2.88	5.15	-22.9	



3.5 DEP.NAT(254NM) (UE/L) UNCLEAN

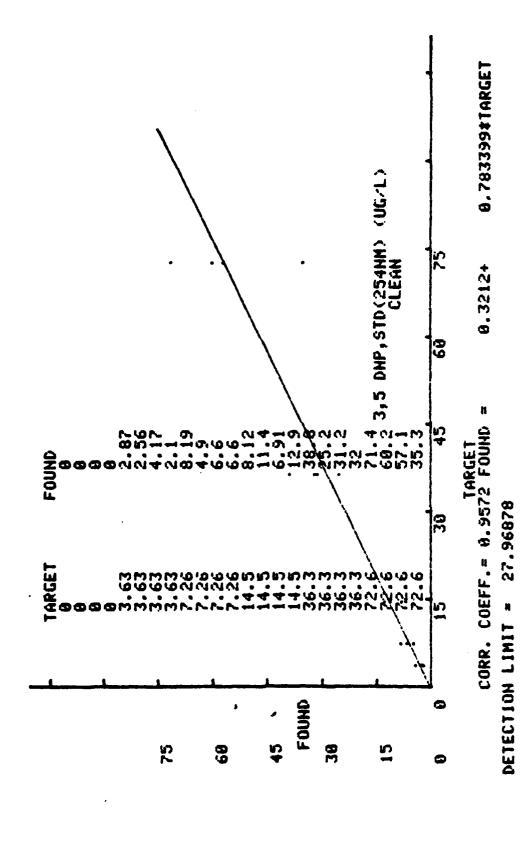
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	a. aa aa	ۥ0000	
3.63	1.74	1.60	3.91	2.64	
7.26	€.75	3.80	6.30	3.30	
14.5	7.48	7.45	19.3	9.60	
36.3	19.4	18.0	35.0	22.0	
72•6	61.0	42.3	64.2	32.3	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0333	0.0000	0.000	0.0000	0.60 P	
3.63	2.25	8 • 686	30∙5	-38-1	
7.26	4.29	1.36	31.7	-40.9	
14.5	11.0	S-65	51.6	-24-4	
36.3	23.6	7.78	33.0	-35∙0	
72.6	49.9	15.2	30.5	-31.2	



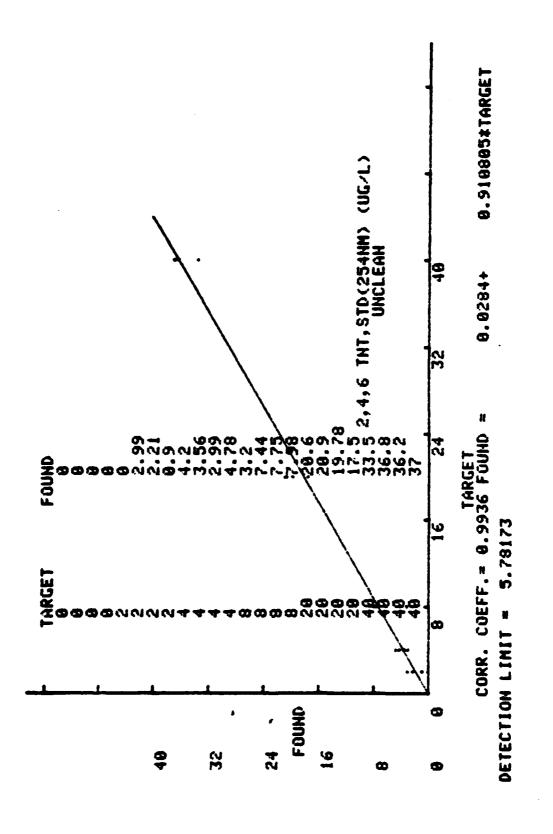
3,5 DNP,STD(254NM)	(UG/L)	CLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4.	
0.0000	0.0000	0.0000	0.000 0	0.0000	
3.63	2.87	2.56	4.17	2.10	
7•26	8.19	4.90	6.60	6.60	
14.5	8.12	11.4	6.91	12.9	
36.3	38.8	25.2	31.2	32.0	
72.6	71.4	60.2	57.1	35.3	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCUPACY
0.0000	0.0000	0.0000	0.0000	0.0000
3.63	2.92	0.888	30.4	-19.4
7.26	6.57	1.34	20.4	-9.47
14.5	9•83	2.79	28.4	-32.2
36.3	31.8	5.57	17.5	-12.4
72.6	56.0	15.1	27.0	-22.c



2.4.6 TNT.STD(254NM)	(UG/L)	UNCLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0009	0.0900	
2.00	0.0000	2.99	2.21	0.900	
4 • 0 0	4.20	3.56	2.99	4.78	
8.00	3.20	7.44	7.75	7.98	
20.0	20.6	20.9	19.8	17.5	
40.0	33.5	36.8	36.2	37.0	

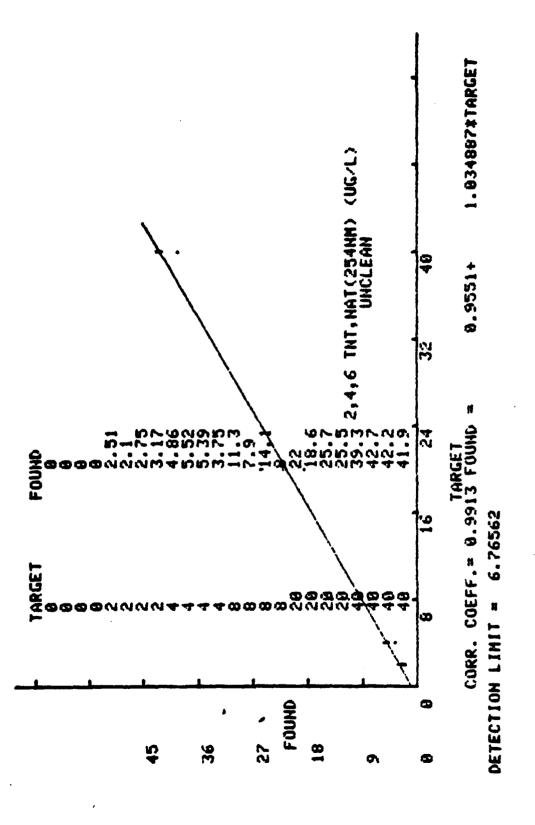
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0. 0000
2.00	1.52	1.33	. 87.4	-23.8
4 • 0 0	3.88	0.776	20.0	-2.94
8 • 0 6	6.59	2.27	34.5	-17.6
20 • C	19.7	1.54	7.81	-1.53
40.0	35.9	1.62	4.51	-10.3



2.4.6 TNT.NAT(254NM) (UG/L) UNCLEAN

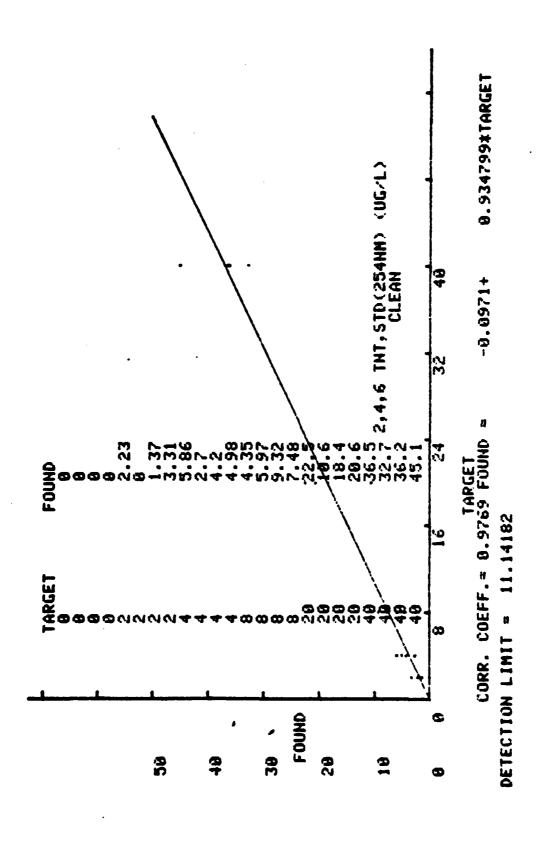
TARGET CONCENTRATION	<u>1</u>	DAY 2	3	4	
0.0020	0.0000	9.0000	0.0000	0.0000	
2.00	2.51	2 • 1 0	2.75	3.17	
4.90	4.86	5.52	5.39	3.75	
8 • 0 C	11.3	7.95	14.1	00.8	
20.0	22.0	18.6	25.7	25.5	
40.ù	39.3	42.7	42.2	41.9	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
2.0063		0.000	0.6000	0.0000	
2.00	2 • 5 3	0.448	17.0	31.6	
4 - 0 0	4.88	0.806	16.5	22.0	
8 • 0 G	10.3	2.97	28.8	29.1	
20.0	22.9	3.36	14.6	14.7	
46.0	41.5	1.52	3.66	3.81	



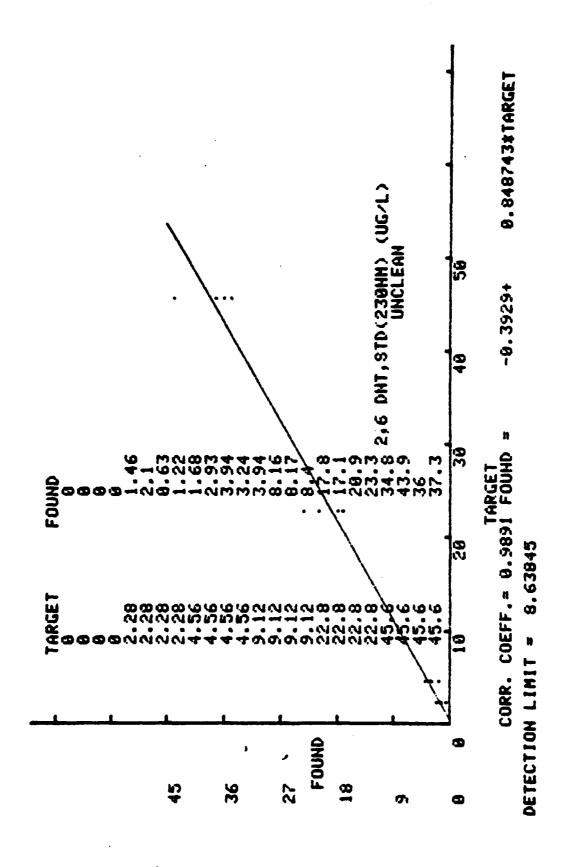
2,4,6 TNT,STD(254NM) (UG/L)	CLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.3000	0.0000	0.000	
2 • 0 0	2.23	0.0000	1.37	3.31	
4.00	5.86	2.70	4.20	4.98	
0.00	4.35	5.97	9.32	7.48	
23.0	22.5	10.6	18.4	20.6	
40.0	36.5	32.7	36.2	45.1	

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0.5000	0.0000	0.0000	0.0000	
2.00	1.73	1 • 4 0,	81.0	-13.6	
4.00	4.43	1.34	30.2	10.9	
8.00	6.78	2.12	31.3	-15.3	
20.0	18.0	5.23	29.0	- 9.88.	
40 • G	37•6	5.27	14.0	-5.94	



2,6 DNT,STD(230NM)	(UG/L)	UNCLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.000	0.0000	0.0000	0.0000	
2.28	1.46	2.10	0.630	1.22	
4.56	1.68	2.93	3.94	3.24	
9.12	3.94	8 • 16	8.17	8 • 4 0	
22.8	17.8	17.1	20.9	23.3	
45.6	34.8	43.9	36.0	37.3	

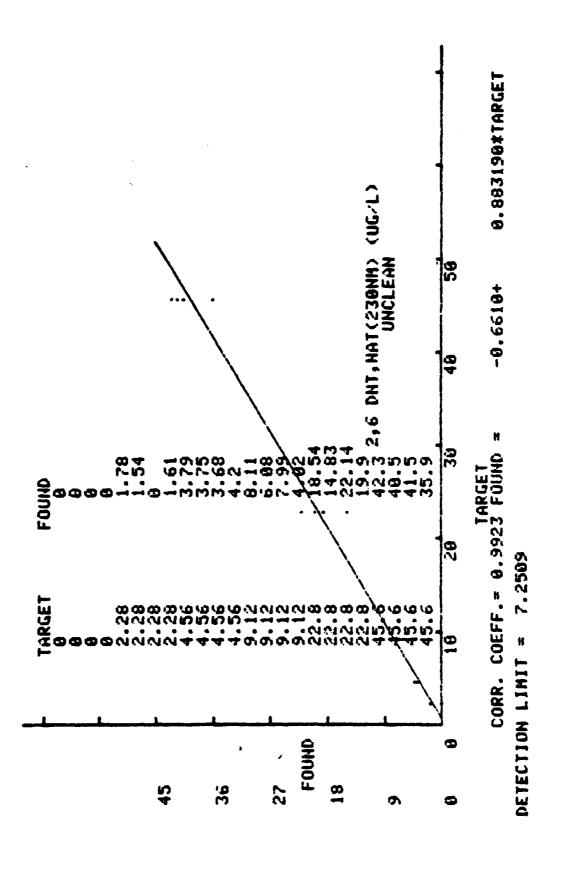
TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.000	0.0000
2 •2 ੪	1.35	0.608	45.0	-40.7
4.56	2.95	0.945	32.1	35.4
9.12	7.17	2.15	30.1	-21.4
22.6	19.8	2.87	14.5	-13.3
45.6	38.0	4.06	10.7	-16.7



2.6	DNT . NAT	(230NM)	(UG/L)	UNCLEAN
_ , ,	D 10 1 3 10 - 1	4 2 0 0 14117		011666711

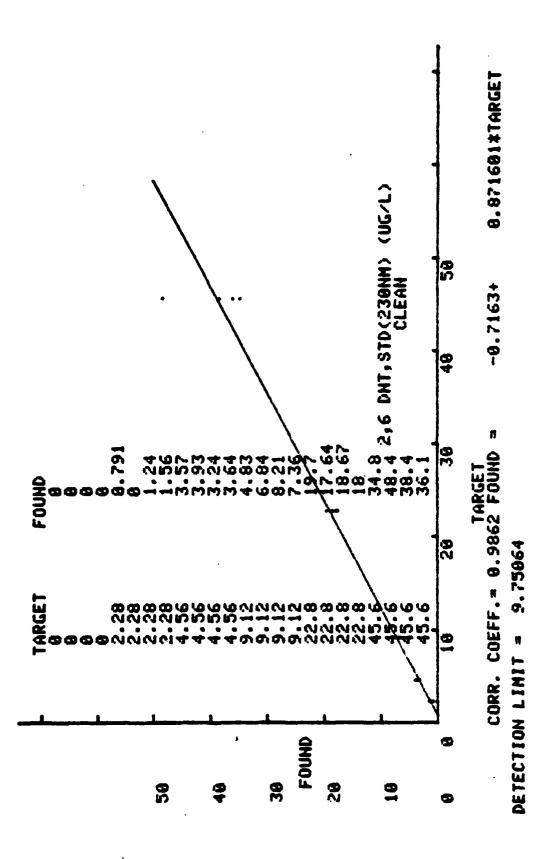
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	9.0099	0.0000	0.0000	
2.28	1.78	1.54	0.0000	1.61	
4.56	3.79	3.75	3.68	4.20	
9.12	8.11	6.08	7.99	4.02	
22.8	18.5	14.8	22.1	19.9	
45.6	42.3	40.5	41.5	35.9	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	3 • 6 6 6 9	0.0000	8.6999
2 • 2 8	1.23	0.828	67.2	-45. 9
4.56	3.85	0.234	6.08	-15.5
9.12	6.55	1.93	29.4	-28 +2
22•8	18.9	3.07	16.3	-17.3
45.6	40.0	2.86	7.15	-12.2



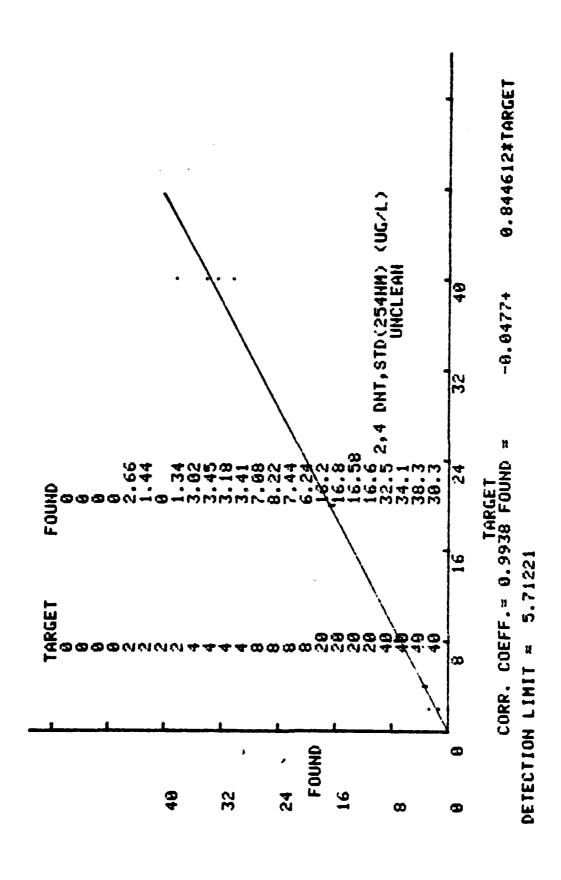
2.6 DAT.STD(230NM) (UFZL)		CLEAN		~~~~~~~	
TARGET COMMENTRATION		DAY ?	7	4	******
0.6000	a.√uger	0.6000	ប ្ បានបំខ	0.0000	
2.25	0.791	0.00	1.24	1.56	
4.56	3.57	₹.°3	3.24	3.54	
9.12	4.83	6.84	8.21	7.36	
22.8	19•7	17.6	18.7	18.0	
45.6	34.8	48.4	38.4	36 • 1	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.5080	0.0000	0.5050
2.26	T.898	2.677	. 75.4	- 60 • 6
4.56	3.5°	0.283	7.88	-21.2
9.12	6.81	1.44	21.1	- 25 • ₹
22.0	18.5	0.905	4.85	-18.8
45•€	39.4	ۥ17	15.6	-13.5



2,4 DNT,STD(254NM)	(UG/L)	UNCLEAN			
TARGET CONCENTRATION	1.	DAY 2	3	4	
0.0000	0.0000	0.000	0.0000	0.0000	
2.00	2.66	1.44	0.000	1.34	
4.53	3.92	3 • 45	3.18	3.41	
8.00	7.98	8.22	7.44	6.24	
20.0	16.2	16.8	16.6	16.6	
40.3	32.5	34.1	38.3	30.3	

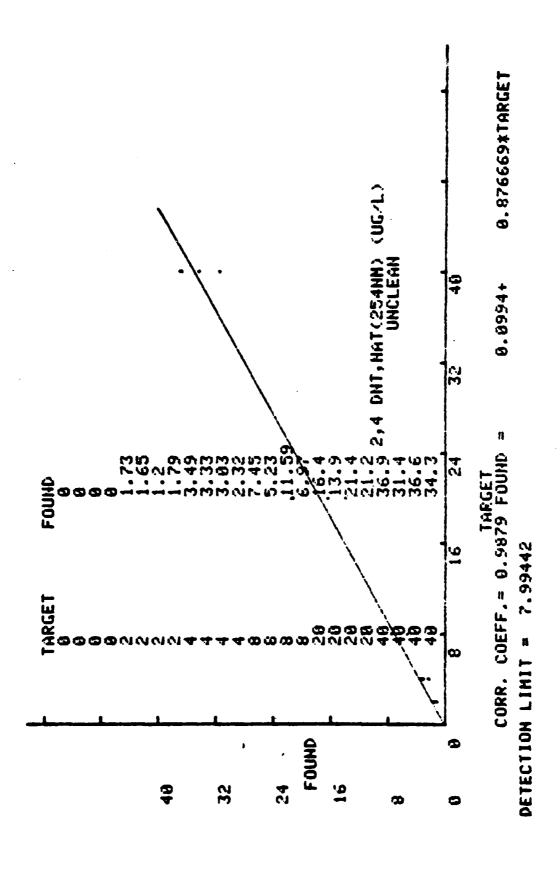
TARGET CONCENTRATION	AVFRAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PEPCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
2.55	1.36	1.09	. 79.9	-32.0
4 • 6 0	3.26	0.202	6.19	-18.4
8 • 0 0	7 • 2 4	0.822	11.3	-9.44
20.0	16.5	0.251	1.52	-17.3
46.0	33.8	3.38	10.0	-15.5



ZYT DNIJNAILZJTNNI LOUPET - UNCLEAN	2,4	DNT . NAT (254NM) (UG/L)	UNCLEAN
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TARGET CONCENTRATION	1	DAY 2	3	4	
0.000.0	0.0000	6.0000	0.0000	0000	
2 • 0 0	1.73	1.65	1.20	1.79	
4 • 0 9	3.49	3.33	3.3	2.32	
8.00	7.45	5.23	11.6	6.97	
20.0	16.4	13.9	21.4	21.2	
40.0	36.9	31.4	36.6	34.3	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD Deviation	PERCENT Imprecision	PERCENT Inaccuracy
9.0030	0.1000	0.0000	0.0000	0.0000
2.90	1.59	0.268	16.8	-2C • 4
4.09	3.04	0.518	17.0	-23.9
8.00	7.81	2.69	34.5	-2.30
2 u • 0	18.2	3.78	20.3	=8•8 8
40.0	34.8	2.55	7•32	-13.0

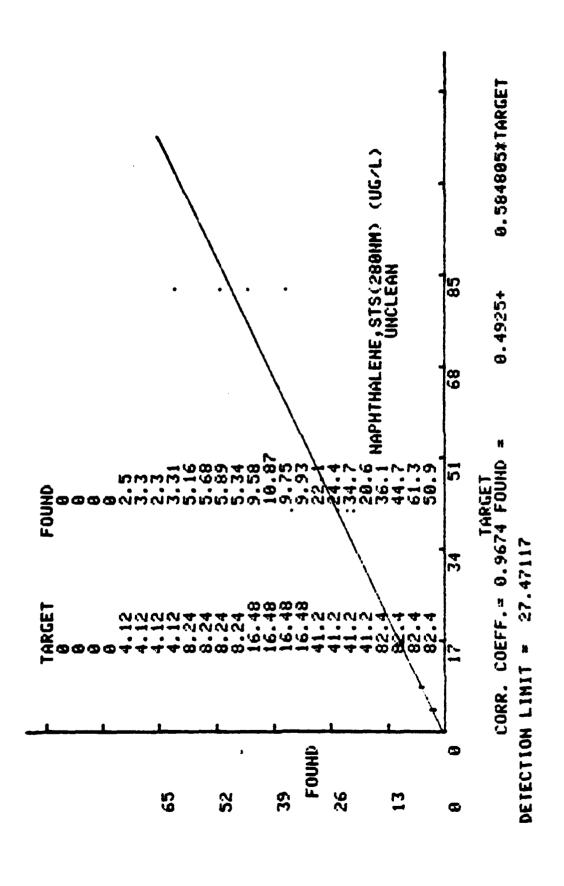


2.4 DNT.STD(254NM)	(UG/L)	CLEAN			
TARGET CONCENTRATION]	DAY 2	3	4	7 P & 7 B &
0.0000	0.0000	0.0000	9.0000	0.0000	
2.00	1.86	0.0000	0.7000	1.62	
4.00	3.13	2.91	3.70	1.81	
8.00	7.51	6.31	5.46	5.92	
20.0	18.5	16.5	16.6	16.2	
40.0	31.2	38 .7	30.2	35.4	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0.0000	6.0000	0.3000	9.00 <u>0</u> 0	
2.03	0.879	1 • 9 1,	116	- 56.5	į
4.00	2.71	0.608	22.4	-32.2	
8.05	6.30	0.878	13.9	-21.3	
20.0	17.0	1.04	6.13	-15.2	•
40.0	33.9	3.93	11.6	-15.3	

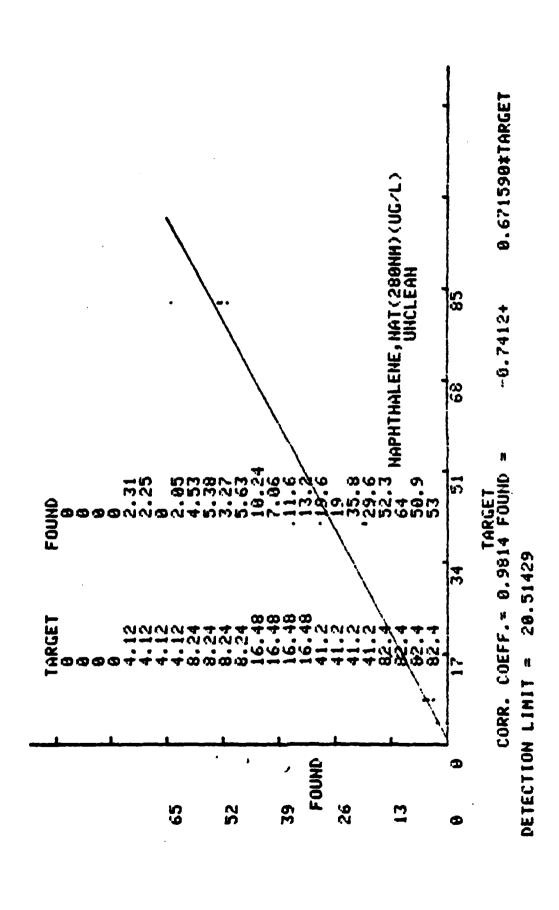
NAPHTHALENE + STD (280NM)	(UG/L)	UNCLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
9.0000	0.0000	0.0000	0.0000	0.0000	
4.12	2.59	3.30	2.30	3.31	
8.24	5.16	5.68	5.89	5.34	
16.5	9.58	10.9	9.75	9.93	
41.2	22.1	24.4	34.7	20.6	
82.4	36.1	44.7	61.3	50.9	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.000	0.0000	0.0000	0.0000
4.12	2.85	0.529	. 18.5	-30.8
8 • 2 4	5.52	0.329	5.96	-33.0
16.5	10.0	0.576	5.74	-39.1
41.2	25 • 4	6.36	25.0	-38.2
82.4	48.2	10.6	22.0	-41.4



NAPHTHALENE, NAT (280MM) (UG/L)		UNCLEAN			
TARGET CONCENTRATION	1	DAY 2	3	Δ	
0.0000	0.0000	0.0000	0.0000	0.000	
4.12	2.31	2.25	0.0000	2.05	
8.24	4.53	5.38	3.27	5.63	
16.5	10.2	7.06	11.6	13.2	
41.2	19.6	19.0	35.8	29.6	•
82.4	52.3	64.0	50.9	53.0	

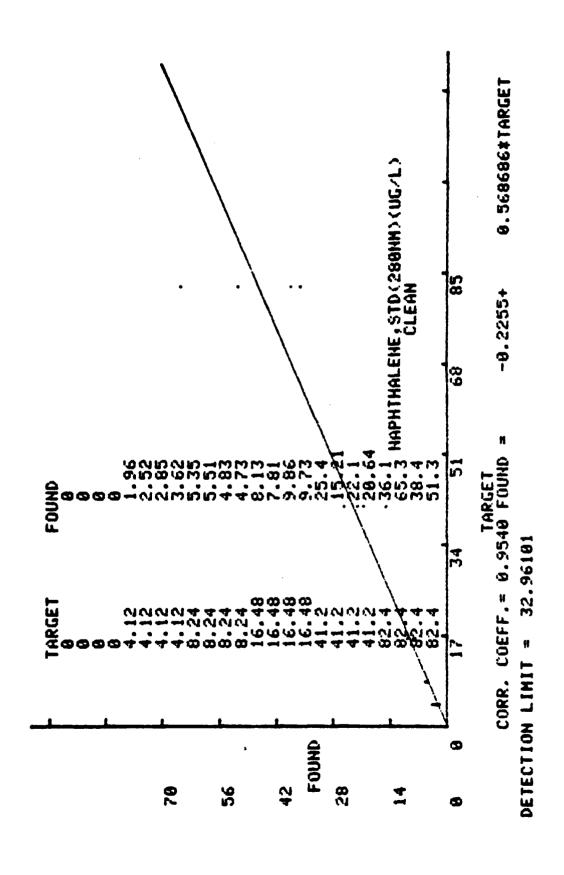
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCUPACY	
0-0000	9.0000	0.0000	0.000	0.0000	
4.12	1.65	1.11	. 67.0	-59.9	
8.24	4.70	1.06	22.6	-42.9	
16.5	10.5	2.61	24.8	-36.1	
41.2	26.0	8.14	31.3	-36.9	
82.4	55.0	6.03	11.0	-33.2	



NAPHTHALENE, STD(290NM) (UG/L) CLEAN					
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.3000	0.0000	0.9000	0.9000	
4.12	1.96	2.52	2.85	3.62	
8.24	5.35	5.51	4.83	4.73	
16.5	8.13	7.81	9.86	9.73	
41.2	25.4	15.2	22.1	20.6	
82.4	36 • 1	65.3	38.4	51.3	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0 • 0 n o o	0.000	0.0000	0.0000
4.12	2.74	0.694.	25.3	- 33•6
8 • 2 4	5.10	0.383	7.50	-38.0
16.5	8.88	1.06	12.0	-46.1
41.2	20.8	4.25	20.4	-49.4
82.4	47.8	13.5	28.2	-42.0

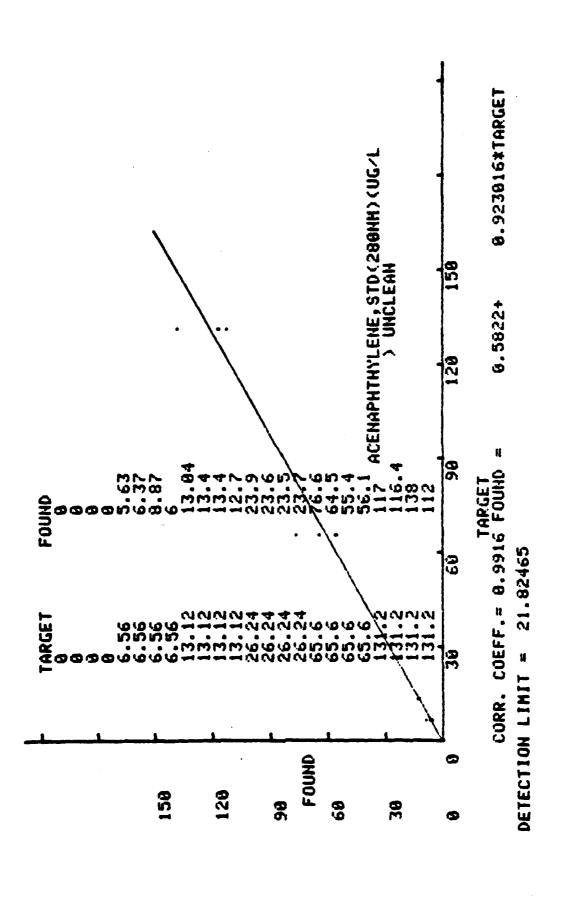
į.



ACENAPHTHYLENE, STD (28 TNM) (UG/L) UNCLEAN

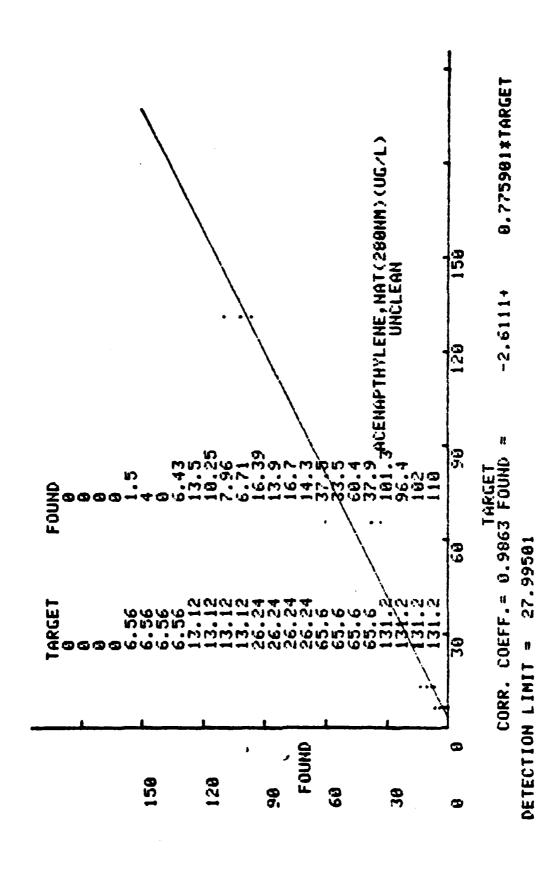
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	0.000	
6.56	5.63	6.37	8.87	6.00	
13.1	13.0	13.4	13.4	12.7	
26•2	23.9	23.6	23.5	23.7	
65•6	76.6	64.5	55.4	56.1	
131	117	116	138	112	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0.0000	0 • 0 0 0 0	0.0000	0.0000	
6.56	6.72	1.47	. 21.8	2.40	
13.1	13.1	0.336	2.56	0.114	
26.2	23.7	0.171	0.723	-9.78	
65•6	63.1	9.87	15.6	-3.73	
131	121	11.6	9.64	-7.89	



ACENAPTHYLENE + NAT (280NM) UNCLEAN							
TARGET CONCENTRATION	· ·	DAY 2	3	4			
0.0000	0.000	0.0000	0.0000	6.0000			
6.56	1.50	4.00	C • 0 0 0 0	6.43			
13.1	13.5	19.3	7.96	6.71			
26.2	16.4	13.9	16.7	14.3			
65.6	37.5	33.5	60.4	37.5			
131	181	96.4	102	110			

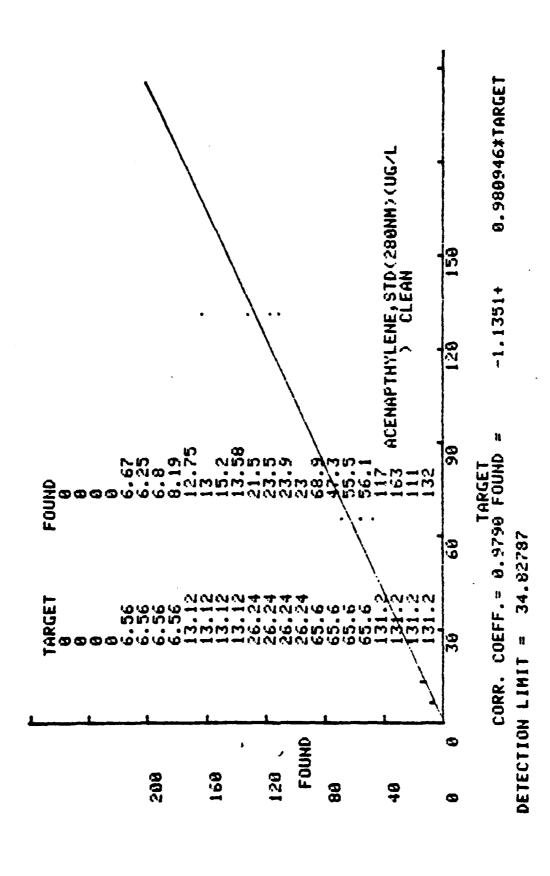
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	2.0000	0.0000	0.0000	0.0000
6.56	2.98	2.83	. 94.9	-54.5
13+1	9•60	2.98	31.8	-26 • 8
26.2	15.3	1.43	9.31	-41-5
65.6	42.3	12.2	28.9	-35.5
131	1 (2	5.63	5.50	-21.9



ACENAPTHYLENE STD (289NM) (UG/L) CLEAN

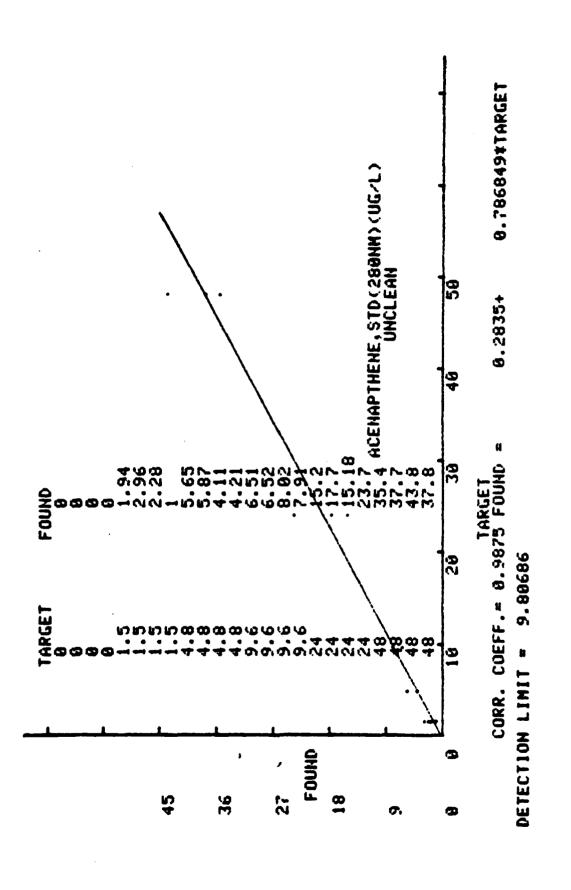
TARGET CONCENTRATION	!	DAY 2	3	4	
0 0 0 0 0	0.0000	0.0060	0.3000	2.000C	
6.56	6.67	6.25	ۥ80	2.19	
13.1	12.8	13.0	15.2	13•€	
26.2	21.5	23.5	23.9	23.0	
65•6	68.9	47.3	55.5	56.1	•
131	117	163	111	132	;

TARGET CONCENTRATION	AVERAGE FOUND VALUE	_	PERCENT IMPRECISION	PERCENT INACCUPACY
0.0000	1 • 00 9 G	0-0000	0.0000	0.0000
€ •5€	5.58	C.842	12.1	6.36
13-1	13.6	1.19	8 • 58	3.91
26.2	23.0	1.05	4.57	-12 • 4
65.6	56.9	8.92	15.7	-13-2
131	131	23.2	17.8	-0.343



ACENAPTHENE , STD (260NM)	(UG/L)	UNCLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.9000	9.• 0 0 0 0	9.0000	0.0000	0.0000	
1 • 5 0	1.94	2.96	2.28	1.000	
4.83	5.65	5.87	4.11	4 • 2 1	
9.60	6.51	6.52	8.02	7.91	
24.0	15.2	17.7	15.2	23.7	
48.0	35.4	37.7	43.8	37.8	

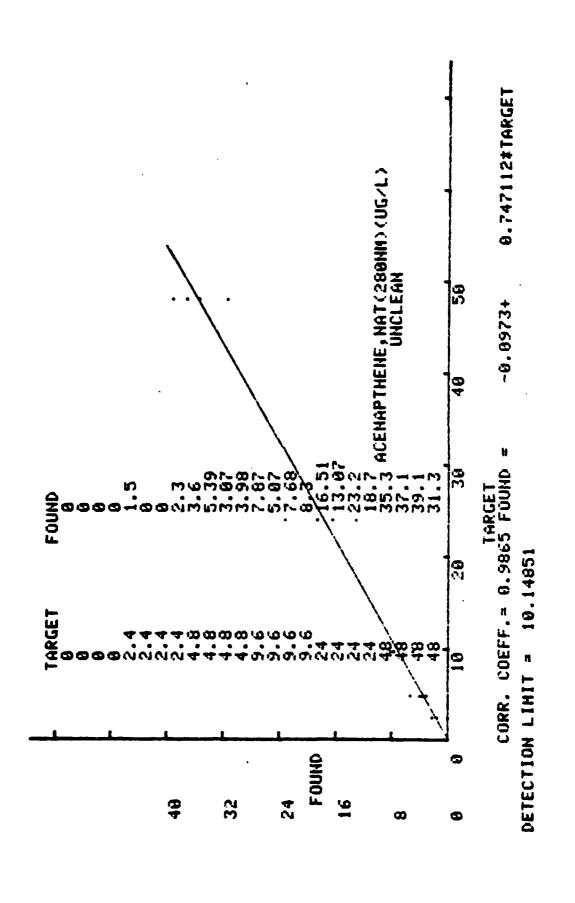
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCUPACY
0.0000	0.0000	0.0000	0.0000	0.000
1.50	2.04	0.816	. 39.9	36.3
4.80	4.96	0.929	18.7	3.33
9•60	7.24	0.838	11.6	-24.6
24.0	17.9	4.01	22.4	-25.2
48.0	38.7	3.59	9 .29	-19•4



ACENAPTHENE , NAT (200NM) (UG/L) UNCLEAN

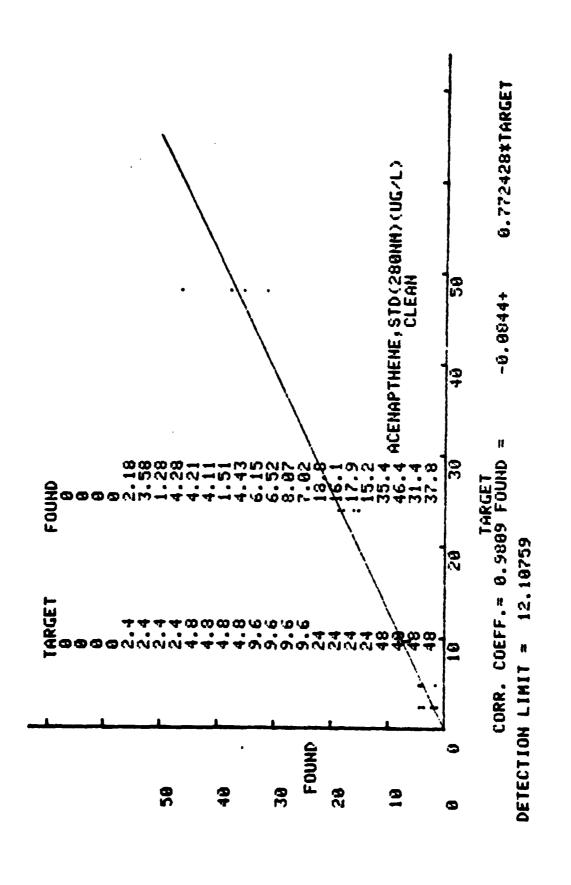
TARGET CONCENTRATION	1	D A Y	3	4	
0.000	0.0000	0.0000	0.0000	0.0000	
2.43	1.50	0.0000	0.0000	2.30	
4.85	3.60	5.39	3.37	ىرىن. 3	
9.60	7.87	5 • 17	7.68	8.30	
24.0	16.5	13.1	23.2	18.7	
48.0	35.3	37.1	39.1	31.3	

TARGET CONCENTRATION	AVERAGE Found value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT Inaccupacy	_
G • O C O B	0.000	0.000	0.0000	0.0000	
2.40	0.950	1.14	120	-60.4	
4 • 8 1	4.01	0.993	24.8	-16.5	
9.60	7.23	1.46	20.2	-24.7	
24.0	17.9	4.24	23.7	-25.5	
48.0	35 • 7	3.32	9.30	-25.6	



ACENAPTHENE + STD (28	CLEAN				
TARGET CONCENTRATION	1	DAY	3	4	; ;
0.0000	C • 0 C O U	0.3090	0.0000	0.0000	:
2 • 4 0	2.18	3.58	1.28	4.28	,
4.80	4 • 21	4.11	1.51	4 • 4 3	
9.60	6.15	6.52	8.07	7.02	:
24.0	18.8	16.1	17.9	15.2	í
48.0	35.4	46.4	31.4	37.8	

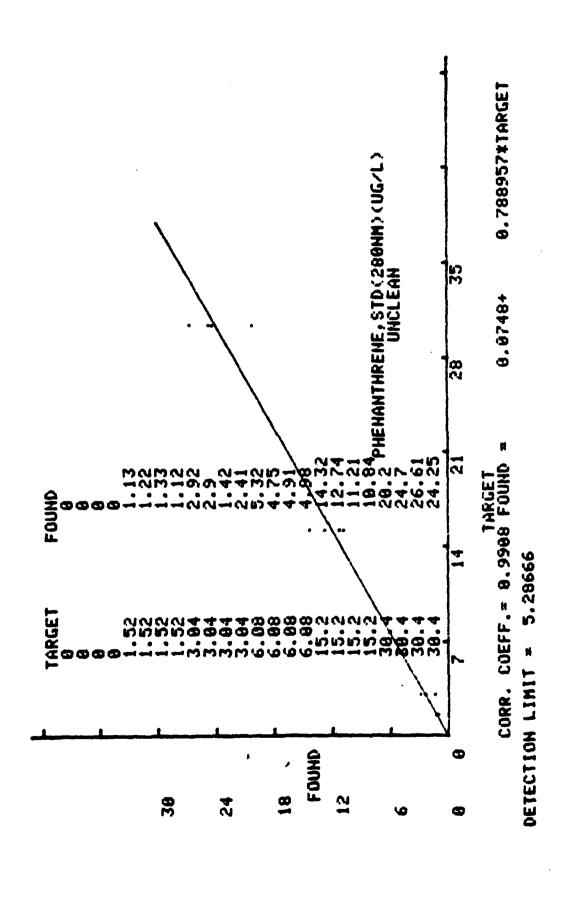
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
C.600ò	9.0000	0.0000	0.0000	0.2000	{
2 • 4 0	2.83	1.35.	47.8	17.9	į
4.80	3.5 <i>6</i>	1.38	38.6	-25.7	ì
9•60	6.94	0-833	12.0	-27.7	
24.0	17.0	1.64	9.67	-29.2	ï
48.0	37.7	6.34	16.8	-21.4	



PHENANTHRENE, STD (280NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0090	0.0000	0.0000	
1.52	1.13	1.22	1.33	1.12	
3 • 0 4	2.92	2.90	1.42	2 • 4 1	
6.08	5.32	4.75	4.91	4.98	
15.2	14.3	12.7	11.2	10.8	
30.4	20.2	24.7	26•6	24.3	

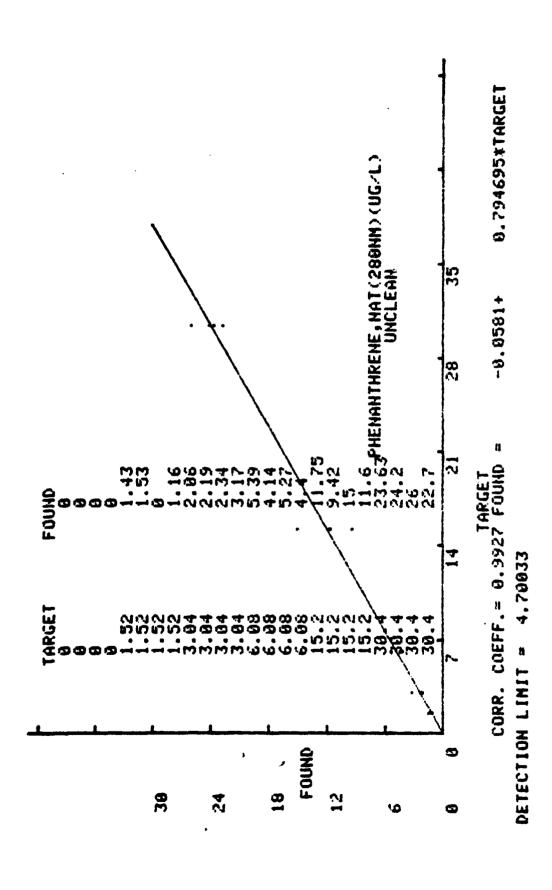
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0 . 0 0 0 0	0.000	0.0000	5.5000
1.52	1.20	0.0976	. 8.14	-21.1
3.04	2 • 41	0.702	29•1	-20.6
6.08	4.99	0.240	4.81	-17.9
15.2	12.3	1.59	13.0	-19.2
39.4	23.9	2.70	11.3	-21.3



PHENANTHRENE . NAT (280NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	9 . 0 9 3 0	
1.52	1.43	1.53	0.0000	1.16	
3.04	2.36	2.19	2.34	3.17	
80.0	5.39	4.14	5.27	4.40	
15.2	11.8	9.42	15.0	11.6	
30.4	23.6	24.2	26.0	22.7	

TARGET CONCENTRATION	AVFRAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.000	0.000	0.3600	0.6000
1.52	1.03	9.704	68.4	-32.2
3. 4	2.44	0.500	20.5	-19.7
6.08	4.80	0.623	13.0	-21.1
15.2	11.9	2.30	19.3	-21.4
3 C • 4	24.1	1.39	5.76	-20.6



TARGET DAY CONCENTRATION 0.0000 0.5050 0.0000 0.0000 0.0000 1.52 1.60 1.41 1.09 1.77 3.04 2.40 3.40 2.56 2.35 6.38 4.41 4.19 5.32 5.03

12.4

27.4

10.8

28.2

11.3

21.5

12.2

24.8

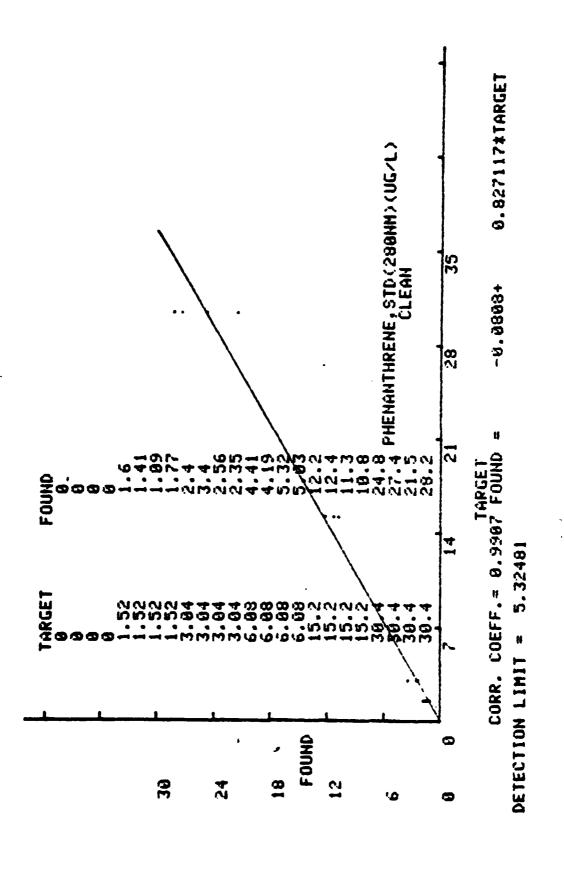
CLEAN

PHENANTHRENE + STD (280NM) (UG/L)

15.2

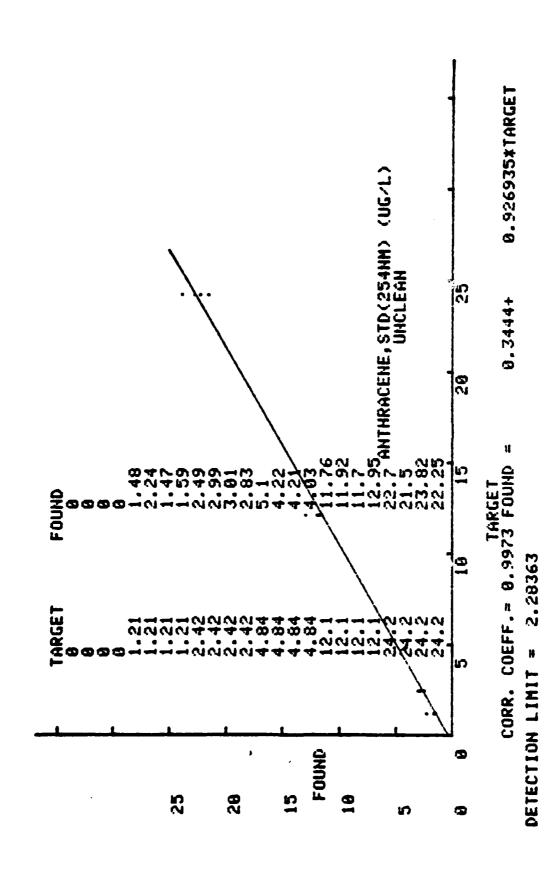
30.4

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0.000	0.0000	0.9000	0.0000	
1.52	1.47	0.291	19.9	-3.45	
3 • 0 4	2.68	0.490	18.3	-11.9	
6.08	4.74	0.527	11.1	-22.1	
15.2	11.7	0.754	6.46	-23.2	
30-4	25.5	3.02	11.9	-16.2	



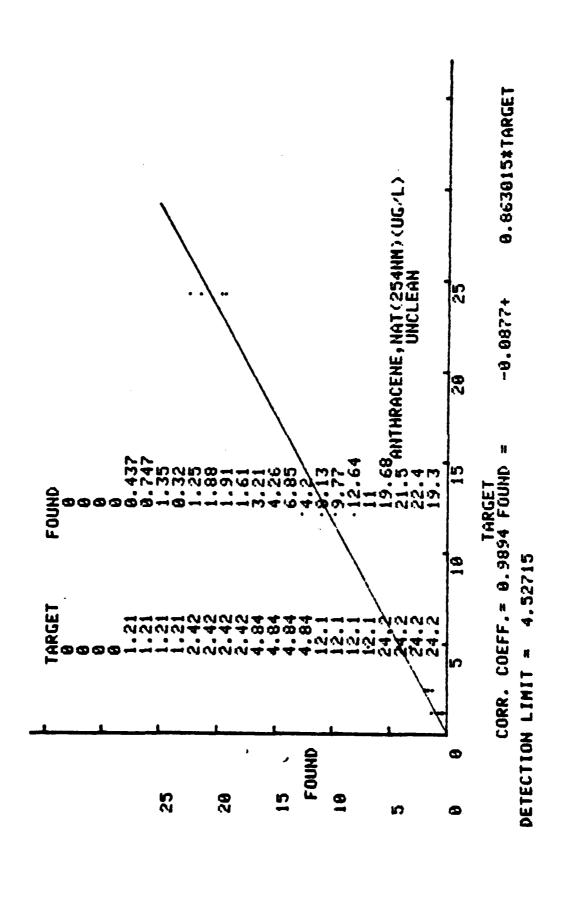
ANTHRACENE .STD (254NM)	(UG/L)	UNCLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.000	0.0000	0.0900	0.0000	5 . 7000	
1.21	1.48	2.24	1.47	1.59	
2.42	2.49	2.99	3.81	2.83	
4.84	5.10	4.22	4 • 21	4.03	
12.1	11.8	11.9	11.7	12.9	
24.2	22.7	21.5	23.8	22.3	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	9.3000	0.0000	0.0000	0.000
1.21	1.69	0.367	. 21.7	40 • 1
2.42	2.83	0.241	8.50	16.9
4.84	4.39	9.481	11.0	-°•39
12.1	12.1	0.586	4 • 85	-0-145
24.2	22.6	0.971	4.30	-6.75



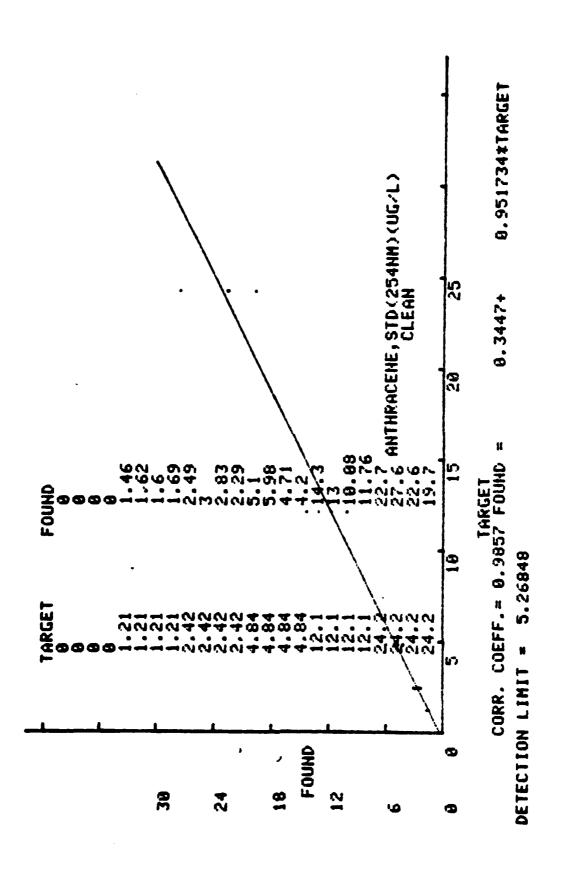
ANTHRACENE (NATUREANM) (UG/L)		UNCLEAN			
TARGET CONCENTRATION		DAY 2	7 ''	4	
0.0000	0.0000	0.000	0.2000	0.0000	
1.21	1.437	0.747	1.35	0.320	
2.42	1.25	1.18	1.91	1 • € 1	
4 - 8 4	3.21	4.26	< •85	4.27	
12.1	0.13	9.77	12.6	11.0	
24.2	19.7	21.5	22-4	19.3	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
9.0000	1.0003	0.000	0.0000	0.0000
1-21	•717	.461	. 64.6	-41.7
2.42	1.66	9.306	18.4	-31.3
4.54	4 • 6 3	1.56	33.6	-4 • Z 4
18.1	10.4	1.91	18.4	-14.2
24.2	20.7	1 • 4 8	7.12	-14.4



ANTHRACENE, STD (254NM) (UG/L)		CLEAN			
T/PGET CONCENTRATION	1	D A Y	3	4	
5.5036	0.0000	0.0000	0.7000	0.0010	
1.21	1.46	1.62	1.60	1.69	
2.42	2.49	3.00	2.63	2.29	
4 • 8 4	5.10	5.98	4.71	4.29	
12.1	14.3	13.0	19.1	11.8	
24.2	22.7	27.6	22.6	19.7	

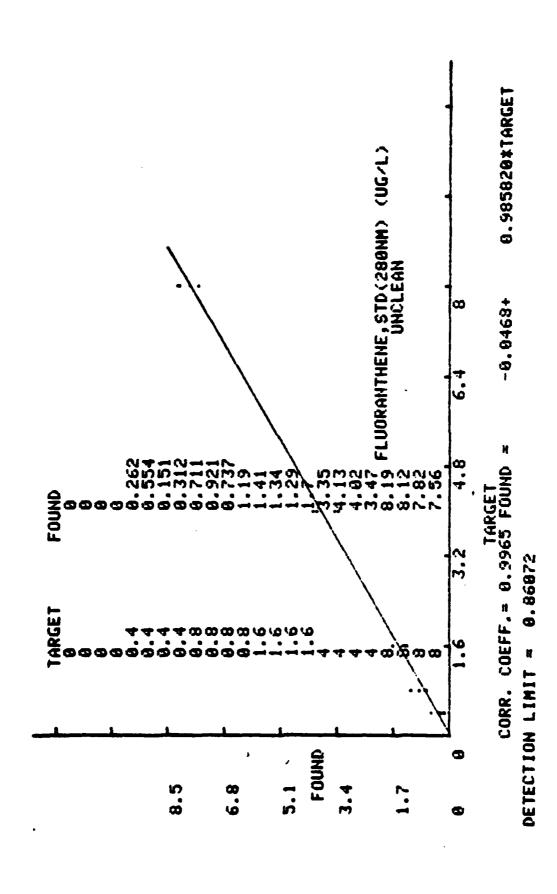
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCUPACY	
0 • C C O O	0 • 0 (3 0	0.0000	0.0000	0.0000	•
1.21	1.59	0.0964	6.05	31.6	
2.42	2.65	0.321	12.1	9.61	
4 • 3 4	5.33	2.752	15.0	3.25	
12.1	12.3	1.80	14.6	1.53	
24.2	23.1	3.28	14.2	-4.34	



FLUORANTHENE STD (280NM) (UG/L) UNCLEAN

TARGET CONCENTRATION	1	D A Y 2	3	4	
0.000	0.0000	9.0000	0.0000	0.0000	
0.439	0.262	0.554	0.151	0.312	
0.860	0.711	0.921	.737	1.19	
1.60	1.41	1.34	1.29	1.70	
4.00	3.35	4.13	4.02	3.47	
8.00	8.19	8.12	7.82	7.56	

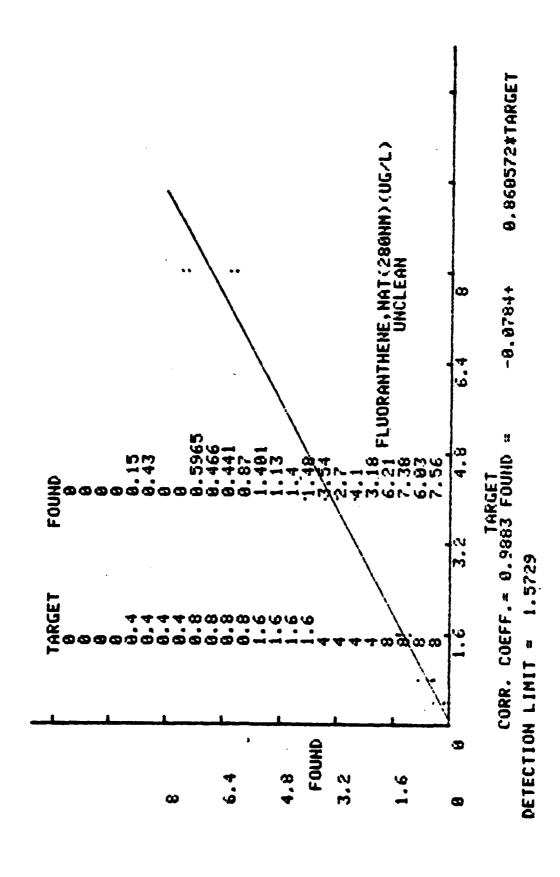
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PEPCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.0000	0.0000	0.0000
0.400	:.325	0.179	53.2	-25.1
0.800	\$• 89 6	3.221	24.8	11.2
1 • 6 5	1.43	0.183	12.8	-10.3
4.70	3.74	0.390	10.4	-6.44
8-00	7.92	0.290	3 • 6 6	-0.969



FLUORANTHENE , NAT (280NM) (UG/L) UNCLEAN

TARGET CUNCENTRATION	1	D A Y	3	4	******
0.0300	0.0300	0.0000	0.0000	0.0000	
0.400	0.15€	0.430	9.0000	0000	
0.800	0.596	0.466	0.441	0.870	
1.60	1.40	1.13	1 • 4 0	1.48	
4 • 0 0	3.54	2.70	4.10	3.18	
8.00	6.21	7.38	6.03	7.56	

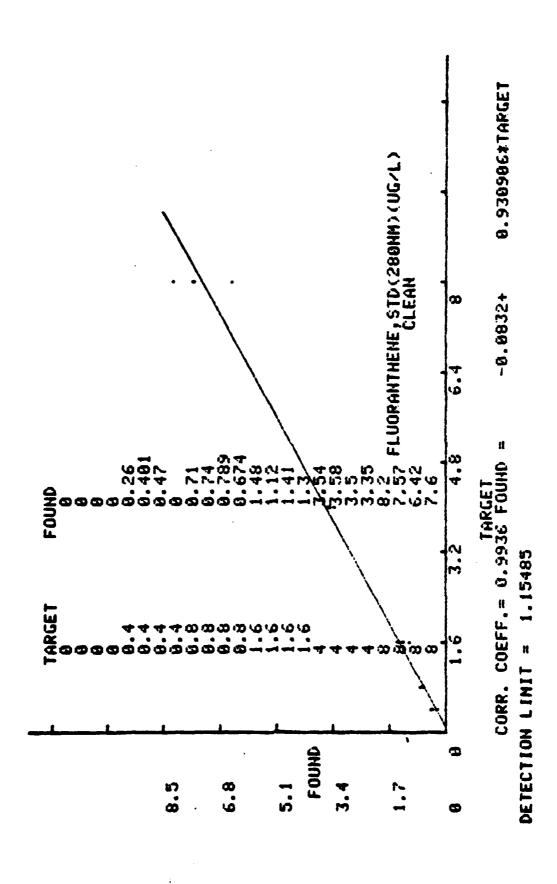
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
6.8066	0 • 0 0 0 0	0.0000	0.000	0.0000
0.400	0.145	0.203	140	-63.8
Ģ•8∶0	0.∙593	0.197	33.1	-25 •8
1.60	1.35	0.153	11.3	-15.5
4 . ū 0	3.38	0.591	17.5	-15.5
8 • 0 0	6.79	0.786	11.6	-15.1



F JORANTHENE + STD (280NM) (UG/L) CLEAN

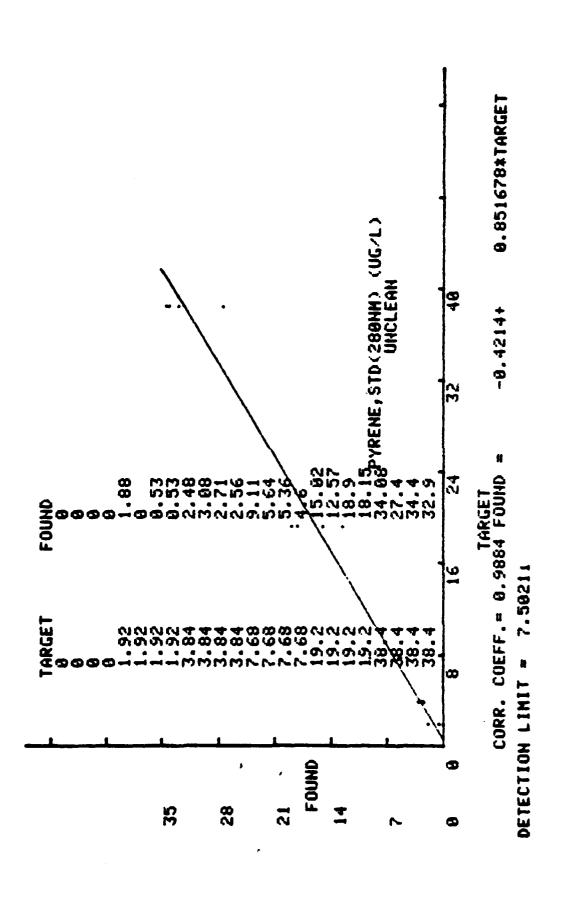
TARGET CONCENTRATION	<u>;</u>	DAY 2	3	4	
0.0000	0.0000	C.0000	0.9800	0.0000	
0.400	0.269	0.401	3.479	*.0009	
0.800	0.710	0.749	0.789	0.674	
1.60	1.48	1.12	1.41	1.30	
4.05	3.54	3.58	3.50	3.35	
8.00	8.20	7.57	6.42	7.60	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT TNACCURACY
0.0000	0.0000	0.000	0.9900	0.7000
0.400	0.4283	0.208	73.5	-29.3
G • 8 G	3 •728	0.0487	6.68	-8.97
1.60	1.33	0.157	11.8	-17+0
4.90	3.49	0.100	2.88	-12.7
8.00	7.45	0.744	9.99	-6.91



PYRENE . STD (280NM)	(UG/L)	UNCLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0600	0.0000	0.0000	0.0000	₹•0000	
1.92	1.88	0.0000	0.530	0.530	
3.84	2.48	3.08	2.71	2.56	
7.68	9.11	5.64	5.36	4.60	
19.2	15.0	12.6	18.9	18.1	
38.4	34.1	27•4	34.4	32.9	

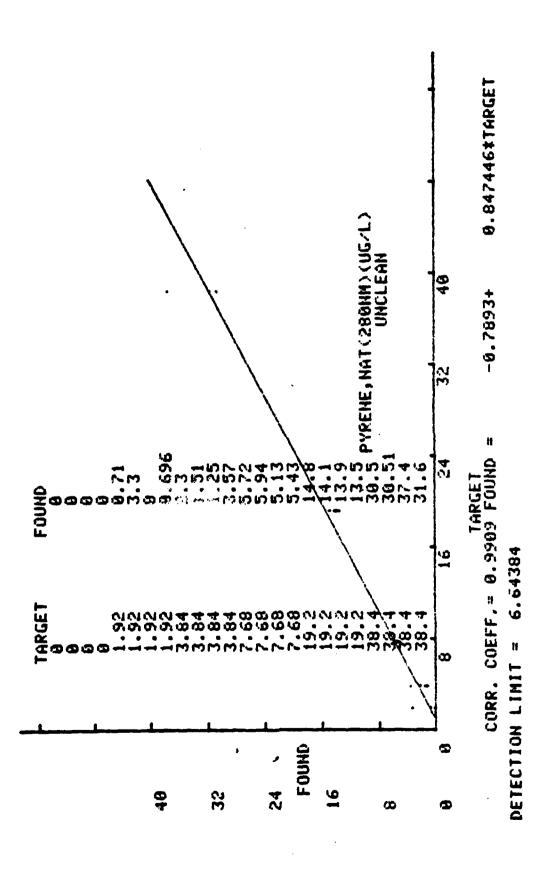
TARGET CONCENTRATION	AVERAGE Found value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.000	0.0000	0.0000	0.0000
1.92	6.735	0.803	. 109	-61.7
3.84	2.71	0.266	9.82	-29.5
7.68	6.18	2.00	32.4	-19.6
19.2	16.2	2.92	18.1	-15.8
38.4	32.2	3.26	10.1	-16.2



PYRENE, NAT (280NM) (UG/L)	UNCLEAN
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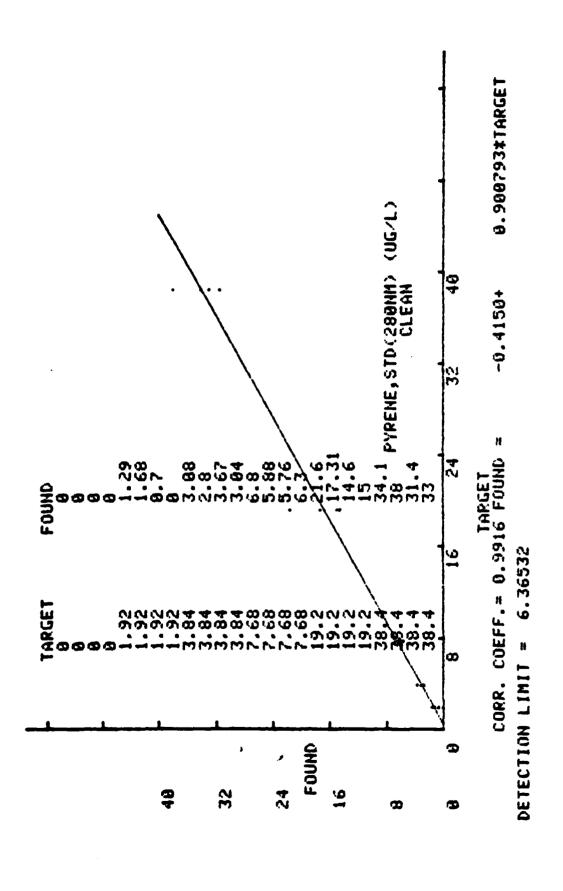
TARGET CONCENTRATION	1	D & Y	7	4	
2.0020	0.0000	0.0000	0.0000	0.0000	
1.92	0.715	3.30	0.0000	0.656	
3.84	2.30	1.51	1.25	3.57	
7 • 6 8	5.72	5.94	5.13	5.43	
19.2	14.8	14.1	13.9	13.5	
38.4	30.5	30.5	37.4	31.6	

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD	PERCENT IMPRECISION	PERCENT
CONCERTRATION	TOUND TALUE	DEALVION	IMPRECISION	14ACCU^ACT
6.9000	0.0000	0.0000	0.0000	0.0000
1.92	1.18	1.45	124	-38.7
3.84	2.16	1.04	48.3	-43.8
7 • 6 8	5.55	0.352	6.34	-27.7
19.2	14.1	3.544	3.86	-26.7
38.4	32.5	3.31	10.2	-15-4



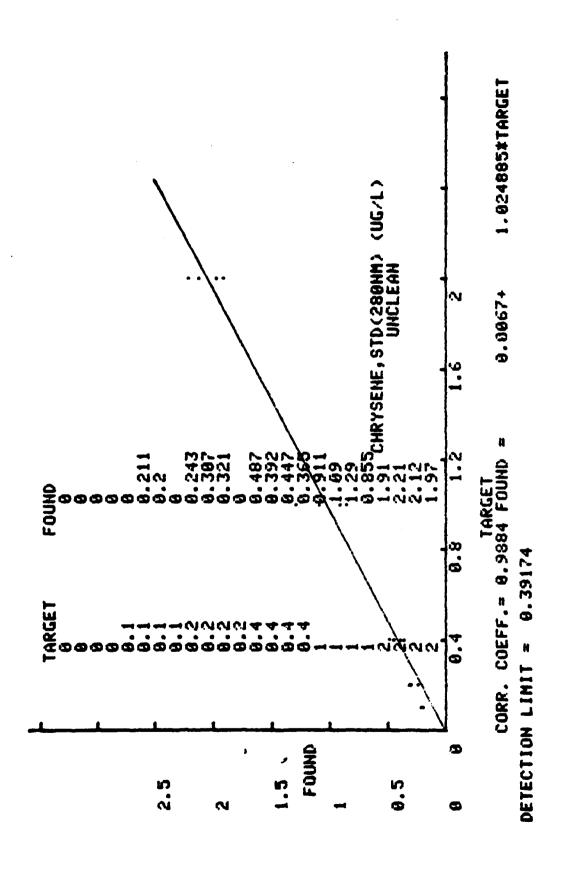
PYRENE + STD (280NM)	(UG/L)	CLEAN		*****	
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0009	C • 0 0 0 0	0.0000	0.0000	
1.92	1.29	1.68	9.799	.0000	
3.84	3.08	2.90	3.67	3 • 9 4	
7.68	6.80	5.88	5.76	6.30	
19.2	21.6	17.3	14.6	15.0	
38.4	34.1	38.9	31.4	33.0	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0.000	0.0000	0.3000	0 • 0 0 0 0	•
1.92	ۥ917	2 .73 2.	79.8	-52.2	
3.84	3.15	0.370	11.7	-18.0	
7.68	6.18	0.471	7.61	-19.5	
19.2	17.1	3.21	18.8	-10 • ×	
38.4	34.1	2.81	8.24	-11.1	



CHRYSENE + STD (28 2NM)	(UG/L)	UNCLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
9.0000	0.0000	0.0000	9.0000	0.0000	
0.1000	0.0000	0.211	0.200	00000	
3 • 2 9 9	0.243	0.307	0.321	0.0000	
0 • 4 0 0	0.487	0.392	0.447	0.365	
1.000	0.911	1.09	1.29	0.855	
2.00	1.91	2•21	2.12	1.97	

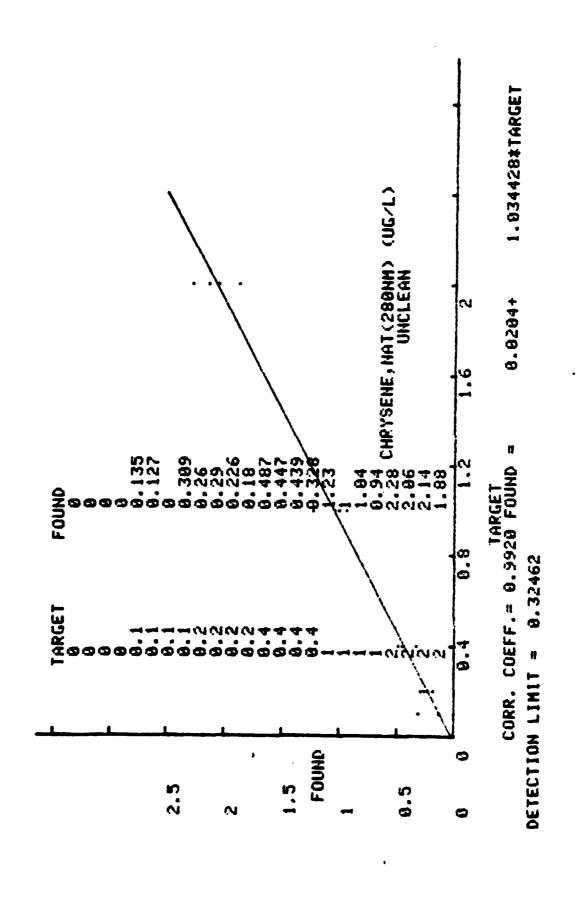
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT Imprecision	PERCENT INACCURACY	
0.0000	0-0000	0.0000	0.0000	0.0000	
0.100	9.103	0.119	. 116	2.75	
0.200	ۥ218	0.149	68.5	್•87	
0.400	0.423	0.3548	13.0	5 • 6 9	
1.00	1.04	0.196	19.0	3.65	
2.00	2.95	0.137	6.68	2.62	



CHRYSENE+NAT(28GNM)(UG/L) UNCLEAN

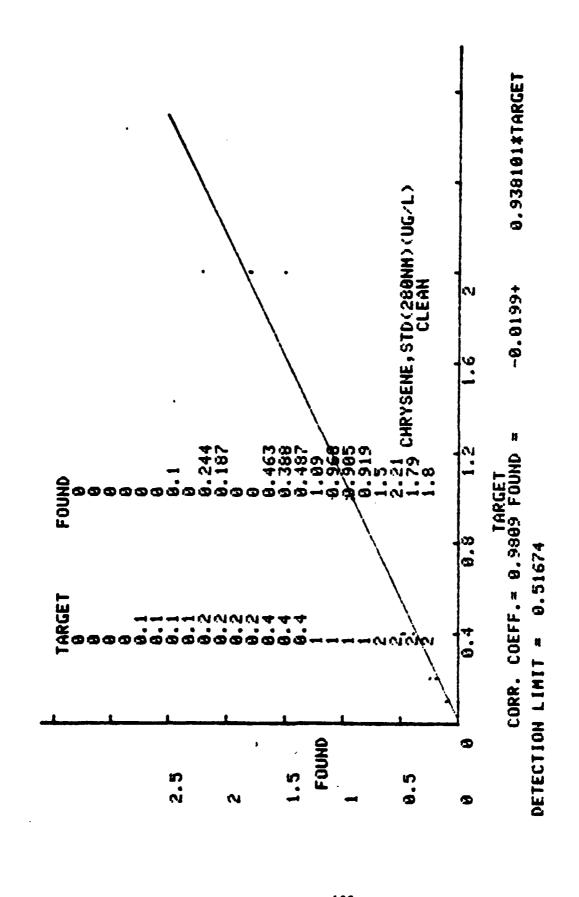
TARGET CONCENTRATION		D A Y 2	3	4	
0.0000	0.0003	0.0000	0.0000	5 • C O C O	
0.1000	0.135	0.127	0.2000	8.309	
0.200	0.260	0.290	0.226	0.180	
C • 4 0 0	€.487	9.447	0.439	0.328	
1 • 0 ė 0	1.23	1.000	1.04	0.949	
2.00	2.28	2.96	2.14	1.88	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0 . 0 0 0 0	0.0003	0.000	9.3000	0.0000
6.196	₹.143	0.127	હ દ• હ	42.7
0.290	2.239	0.0472	19.8	19.5
₹.400	1 • 4 2 5	0.^681	16.9	6.31
1.50	1.05	0.125	11.9	5.25
2.00	2.09	0.167	7.99	4.53



CHRYSENE, STD (280NM) (UG/L)		CLEAN			
TARGET CONCENTRATION	1	DAY			
0.9000	0.0000	0.0000	0.0000	0.0000	
0.1000	0.0000	9.0000	0.1000	0000	
0.200	9.244	0.187	0.0000	0.0000	
0 • 4 © 0	0.463	0.388	0.487	1.09	
1.000	0.968	0.905	0.919	1.50	
2.00	2.21	1.79	1.89	0.0000	

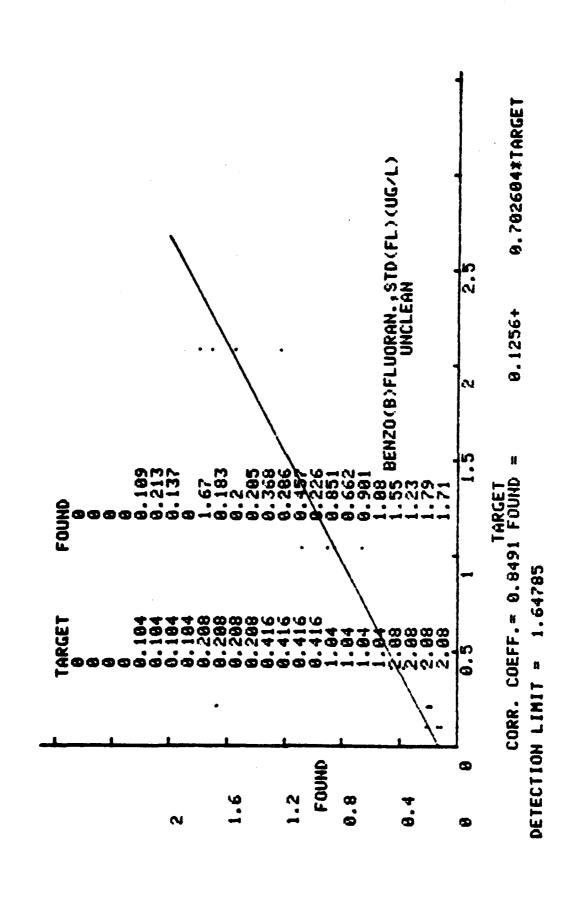
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0008	9.0000	0.0000	6.6660	0.0000
0.100	0.0259	0.0500	200	-75.0
9.200	0.108	0.127	117	-46.1
û . 4 û €	446	0.0516	11.6	11.5
1.60	1.970	0.0841	8.67	-2.95
5 • 0 0	1.82	0.292	16.0	-8.75



BENZO(B)FLUORAN.,STD(FL)(UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	0.0000	
0 - 1 0 4	0.109	0.213	9.137	.0000	
0.208	1.67	0.183	0.210	0.205	
C • 416	0.368	0.286	0.457	0.226	
1.04	0.851	0.662	0.901	1.08	
2.08	1.55	1.23	1.79	1.71	

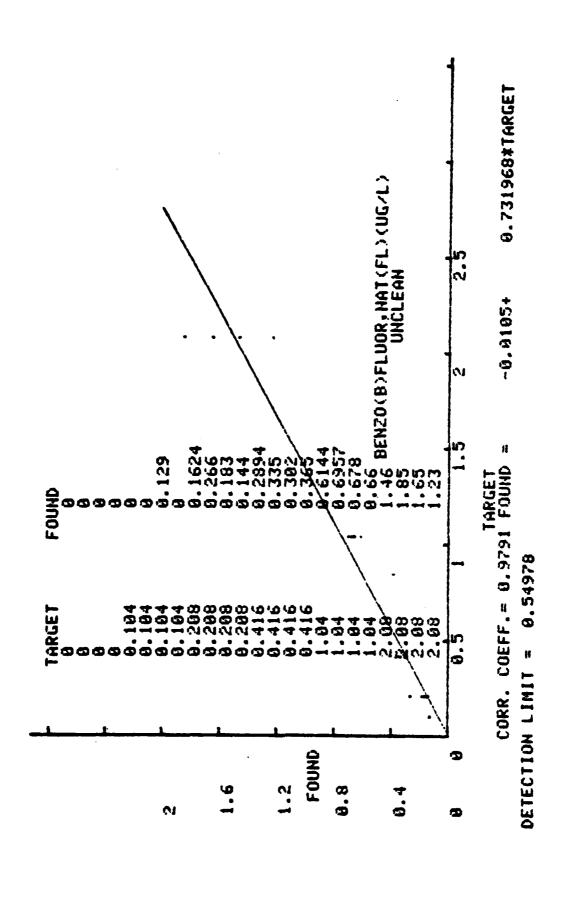
TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT Inaccuracy	
0.0000	0.0000	0.0000	0.0000	0.3000	
0.104	0.115	0.0882	. 76.9	10.3	
0.208	0.564	0.737	131	171	
0.416	0.334	0.100	30.0	-19.7	
1.04	0.873	0.172	19.7	-16.0	
2.38	1.57	0.248	15.8	-24.5	



BENZG(B)FLUOR .NAT (FL) (UG/L) UNCLEAN

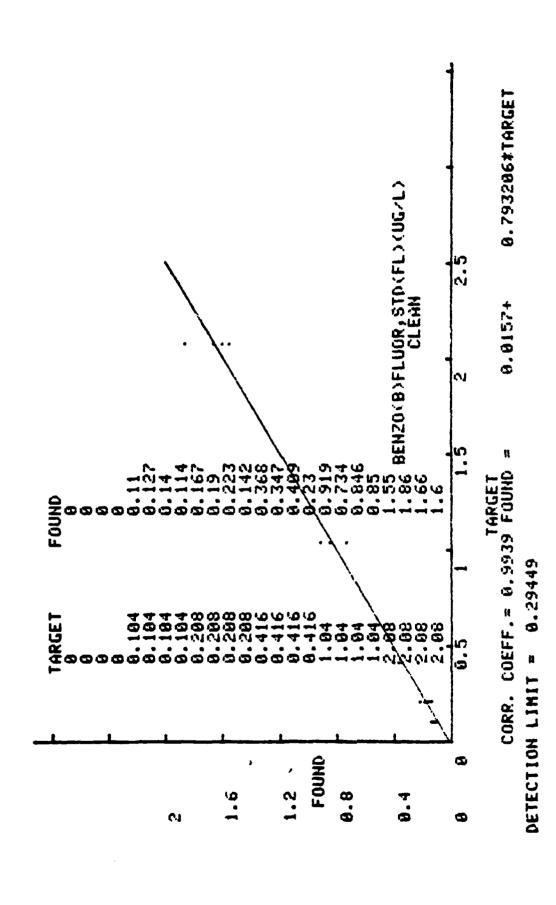
TARGET CONCENTRATION		DAY 2	3	4	
0.8800	0.0000	6.0000	0.9960	9.0500	
9.104	0.0000	.0000	0.129	0.0000	
C.208	0.162	0.266	9.183	C-144	
0.416	0.289	0.335	0.302	0.365	
1.04	0.614	0.696	0.678	0.660	
2.38	1.46	1.85	1.65	1.23	

TARGET CONCENTRATION	AVERAGE Found value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	G.000C	C • 0 0 G O	0.0000	0.0000
0.194	0.0322	0.3645	2 00	-69.0
0.208	0.189	0.0538	28.5	-9.21
9.416	9.323	0.0340	10.5	-22.4
1.34	ۥ662	0.1349	5.29	-36.3
2.06	1.55	0.265	17.1	-25.6



BENZO(B)FLUGR.STD(FL)(UG/L)		CLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.5000	2.0000	0.0000	0.2000	0.0000	
€ •1 € 4	0.110	0.127	0.140	0.114	
≎•208	•167	0.190	0.223	0.142	
0.416	0.369	0.347	0.409	0.230	
1.64	0.919	0.734	0.846	9.850	
2. 08	1.55	1.86	1.66	1.60	

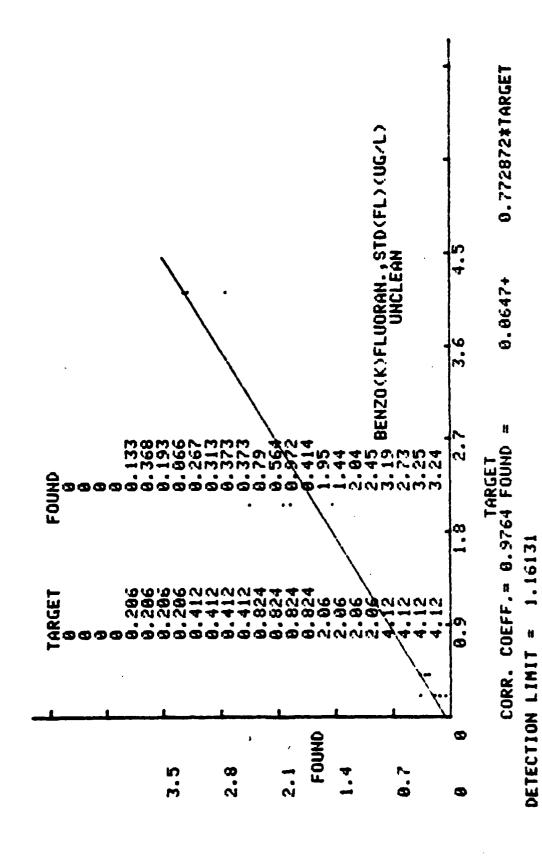
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
9 • 0 6 9 9	0.0000	0.000	9.0000	0 • 0 0 · r
6.104	0.123	0.0136.	11.1	18.5
0.208	5 -189	0.0345	19.1	-13-2
ú•416	0.338	0.0768	22.7	-18.6
1.84	७∙837	0.0766	9.14	-19.5
2.08	1.67	9.136	9.15	-17.8



BENZO(K)FLUORAN., STD(FL)(UG/L) UNCLEAN

TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	0.0000	0.0000	0.0000	
0.206	0.133	0.368	0.193	-0660	
0.412	0.267	0.313	0.373	1.373	
0.824	0.796	0.564	0.972	0.414	
2.06	1.95	1 • 4 4	2.04	2.45	
4.12	3.19	2.73	3.25	3.24	

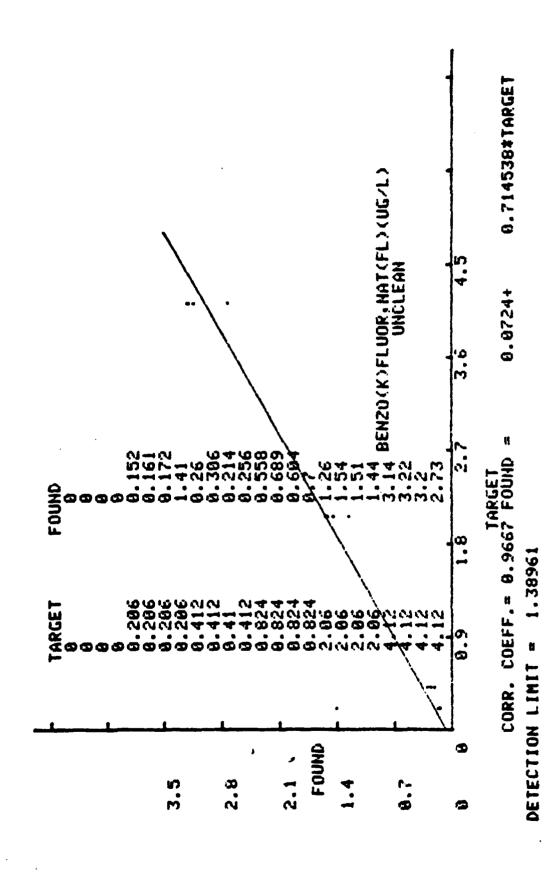
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	-
0.0000	0.000	0.0000	0.0000	0.0000	
0.206	0.190	0.130	. 68•2	-7.77	
·412	0.4331	0.0515	15.5	-19.5	
0.824	° ∙ 685	0.246	35.9	-16.9	
2.06	1.97	0.415	21.1	-4.37	
4.12	3.10	0.250	8.05	-24.7	



BENZO(K) FLUOR . NAT (FL) (UG/L) UNCLEAN

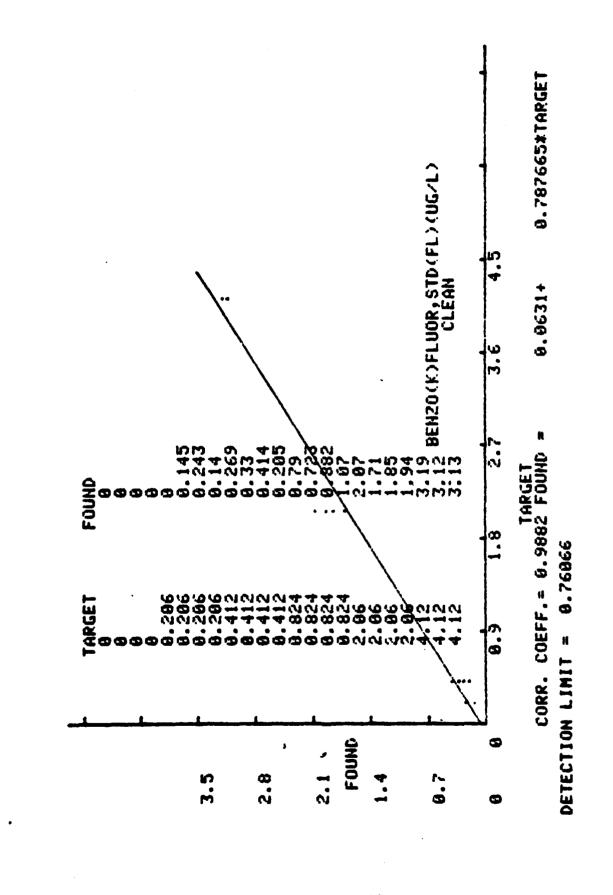
TARGET CONCENTRATION	1	D A Y 2	7	4	
0.0000	3 • 0 0 0 0	0.0000	0.0999	0.0000	•
0.29€	0.152	0.161	0.172	1.41	
÷412	0.260	0.396	1.214	8.256	
€.824	0.558	0.689	0.604	0.700	
2.0€	1.26	1.54	1.51	1.44	
4.12	3.14	3.22	3.27	2.73	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.000	0.0000	0.0000
9•29⊜		0.624 .	132	130
0.412	1.566	0.161	0.0000	-62.6
0 ∙824	J•214	0.0458	0.0000	-47.8
2.36	^•256	0.0655	0.0000	-37.9
4.12		0.0683	10.7	-22.6



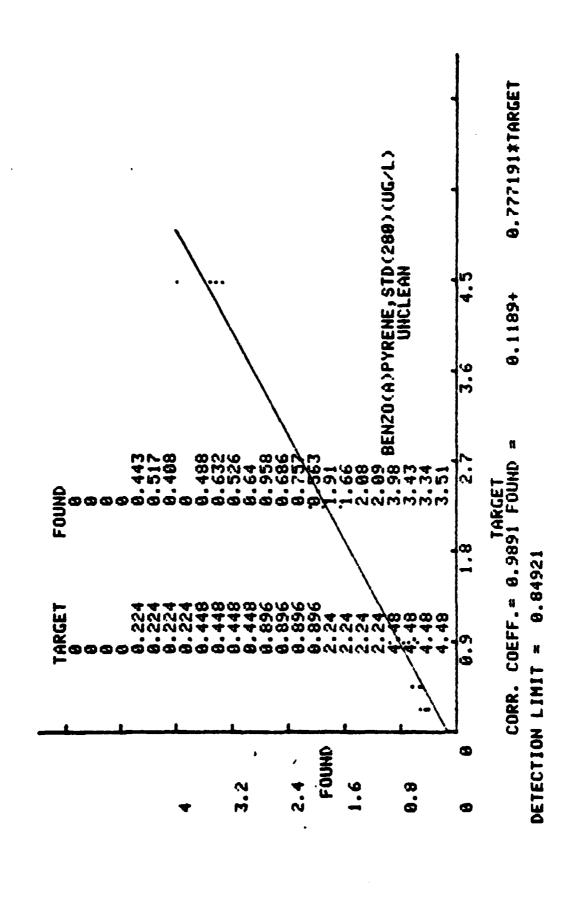
BENZG(K)FLUGR.STD(FL)(UG/L)		CLEAN			
TARGET CONCENTRATION	1	DAY 2	3	4	
0.0000	0.0000	2 • 0 9 2 0	0.0000	0.000	
ũ• 2 ũ6	0.0000	0.145	0.243.	0.140	
C • 412	0.269	0.330	9.414	0.205	
0.824	J.799	0.723	0.882	1.07	
2.06	2.07	1.71	1.85	1.94	i
4.12	3.19	3.12	3.13	0.0000	l

TARGET CONCENTRATION	AVERAGE Found value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
6.0000	0.0000	0.0000	0.2000	0.0000
∂ • 2 0 6	₽•132	0.1000	75.7	-35.9
9.412	0.304	0.0891	29.3	-26-1
0 • 8 2 4	2•866	0.151	17.4	5.13
2.06	1.89	0.152	8.1	-8.13
4.12	3.15	0.0378	1.20	-23.6



BENZO(A)PYRENE STO	UNCLEAN				
TARGET CONCENTRATION	1	DAY 2	3	4	
0.000	0.0000	0.0000	0.0000	0.000	
9.224	8.443	0.517	0.408	1.0938	
0.448	0.488	0.632	0.526	0.640	
0.896	0.958	0.686	0.757	0.563	
2.24	1.91	1.66	2.08	2.09	
4.48	3.98	3.43	3.34	3.51	

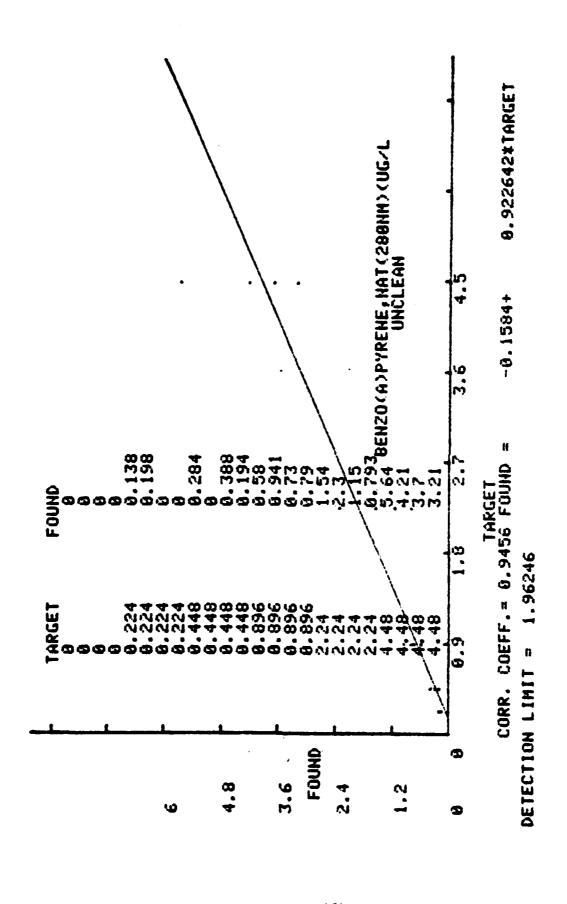
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0 - 0 0 0 0	8 • 0 8 0 0	0.0000
0 • 224	0.342	0.232	68.0	52.7
G - 448	0.571	0.0761	13.3	27.6
9.896	0.741	0.165	22.3	-17.3
2.24	1.93	0.201	10.4	-13.6
4 • 4 8	3.56	0.285	8.00	-20.4



BENZO(A)PYRENE , NAT (280NM) UNCLEAN

TARGET CONCENTRATION	1	5 D A Y	3	4	
0.0000	9.0990	C.0000	0.9000	0.0000	
0.224	0.138	0.198	0.0000	.0000	
0.448	0.284	0.0000	0.388	0.194	•
C.896	0.589	0.941	0.739	0.790	
2.24	1.54	2.30	1.15	0.793	
4.48	5.64	4.21	3.70	3.21	

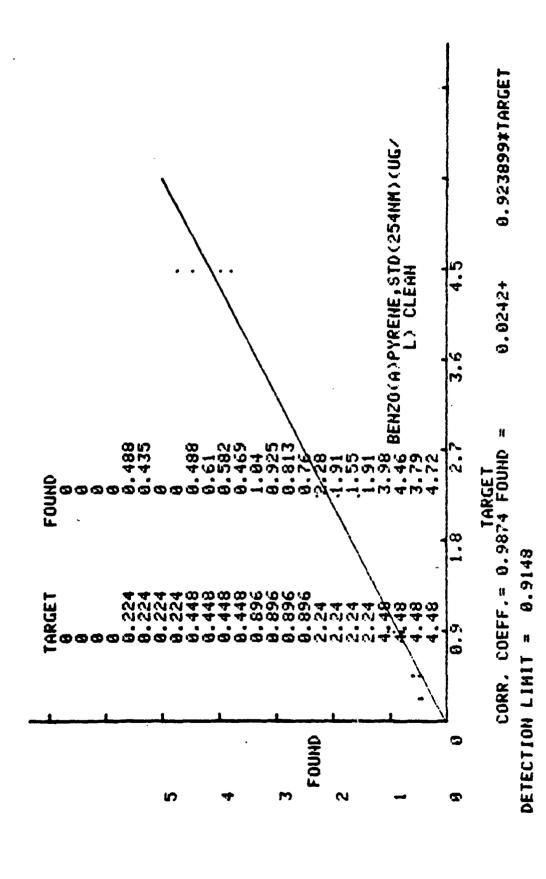
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCUPACY
0.0000	6.0066	0.0000	0.0000	0.0000
0.224	0840	0.100 .	119	-62.5
0.448	9.216	0.165	76.1	-51.7
0.89€	E • 760	0.149	19.7	-15.2
2.24	1.45	0.646	44.7	-35.5
4.48	4.19	1.05	25.0	-6•47



BENZO(A)PYRENE + STD (254NM) (UG/L) CLEAN

TARGET CONCENTRATION	1	2 D A Y	3	4	
0.0000	0.6000	c•0000	.0 • 0 0 0 0	0.0000	
3.224	8.488	0.435	0.0000		
ปิ.448	6.488	0.610	3.582	0.469	,
0.896	1.04	0.925	0.813	0.768	· (
2.24	2.28	1.91	1.55	1.91	:
4 • 4 8	3.98	4.46	3.79	4.72	_

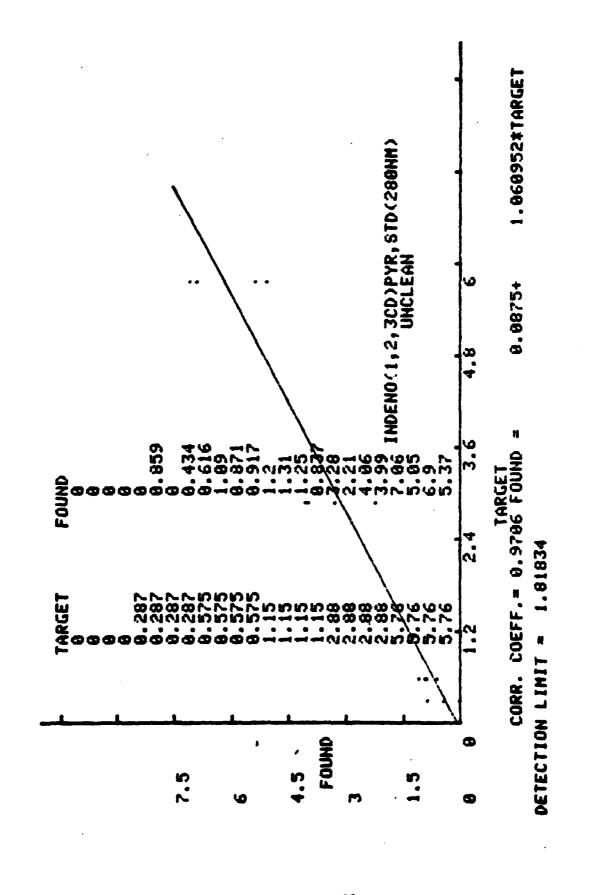
TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY	
0.0000	0.0030	0.0000	0.0000	0.0000	
0.224	.231	0.267	116	3.01	ì
J • 4 4 4	0.537	0.0692	12.9	19.9	{
0 .89 6	ۥ884	0.124	14.1	-1.28	
2.24	1.91	0+298	15.6	-14.6	ĺ
4 • 48	4.24	0.428	10-1	-5.41	Ċ



INDENO(1+2+3CD)PYR++STD(280NM) UNCLEAN

TARGET CONCENTRATION	1	5 DAY	3	4	
0.0600	0.0000	0.9000	0.0000	0.0000	
0.287	0.0000	0.859	0.0000	0.434	
0.575	0.616	1.09	0.871	0.517	
1.15	1.20	1.31	1.25	0.837	
2.88	3.28	2.21	4.06	3.99	
5.76	7.86	5.05	6.90	5.37	

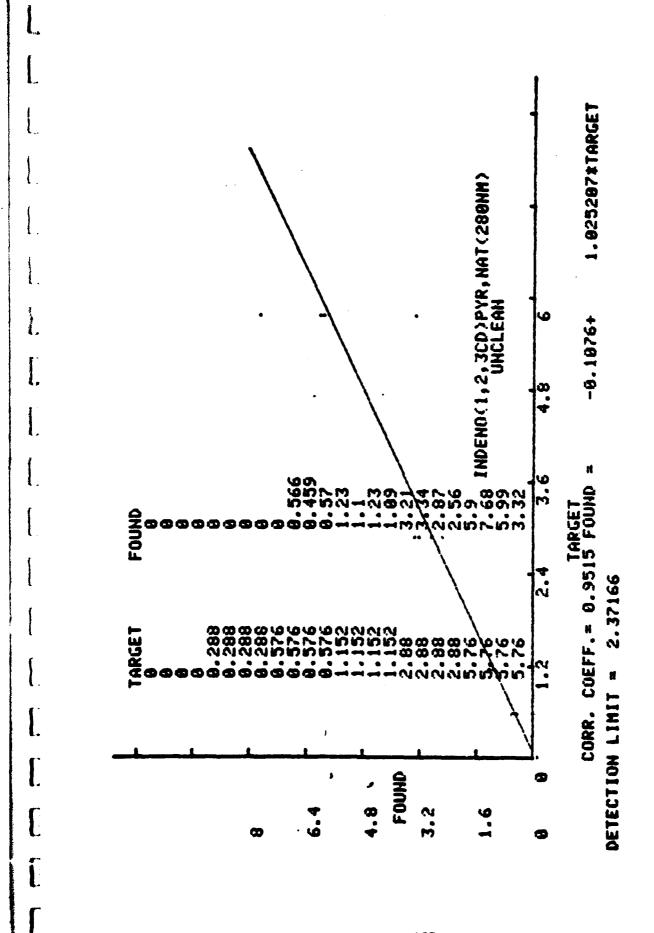
TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCUPACY
0.0000	0.0000	0.000	0.0000	0.000
0.287	0.323	0.412	. 127	12.6
0.575	0.873	0.196	22.4	51.9
1.15	1.15	0.213	18.5	-0.C652
2.88	3.38	0.859	25.4	17.5
5.76	6.09	1.03	16.9	5.82



INDENO(1,2,3CD)PYR,NAT(280NM) UNCLEAN

TARGET CONCENTRATION	1	D A Y	3	4	
0.000C	0.0000	C • 0 0 0 0	0.0000	0.0000	
9.288	0.0000	0.0000	0.2000	.0000	
€.576	0.0000	0.566	0.459	8.579	
1.15	1.23	1.10	1.23	1.00	
2.88	3.21	3.34	2.87	2.56	
5.76	5.9	7•68	5.99	3.32	

TARGET CONCENTRATION	AVERAGE Found Value	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0006	0.0003	0.0000	0.0000	9.0006
J•288	0.0000	0.0000 .	0.0000	-100
1.576	0.399	0.271	67.9	-30-8
1.15	1.16	0.0780	6.71	0-911
2.88	2.99	0.351	. 11.7	3.99
5.76	5.72	1.80	31.4	- 0.651



INDENR(1,2,3CD)PRY,STD(289NM)(UG/L) CLEAN

TARGET CONCENTRATION	1	DAY 2	3	4	
0 0 0 0 0 0	6.0000	0.0000	0.0000	2.0000	
0.287	C.710	0.6300	9.300	.0000	
0.575	0.615	0.512	9.672	0.579	
1.15	1.84	0.903	1.52	0-931	
2.68	3.53	2.73	2.69	3.27	
5.76	7.06	8.07	5.90	5.20	

TARGET CONCENTRATION	AVERAGE FOUND VALUE	STANDARD DEVIATION	PERCENT IMPRECISION	PERCENT INACCURACY
0.0000	0.0000	0.000	0.0000	0.0000
0.287	9.252	0.336.	133	-12.0
.575	0.592	0.0679	11.5	3.00
1.15	1.30	0.460	35.4	12.9
2.68	3.05	0.413	13.5	14.0
5.76	6.56	1.27	19.3	13.8

